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To the Graduate Council:

I am submitting herewith a thesis written by Kyle James Schlachter entitled "Macroscopic Sedimentary Charcoal as a Proxy for Past Fire in Northwestern Costa Rica." I have examined the final electronic copy of this thesis for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Master of Science, with a major in Geography.

Sally P. Horn, Major Professor

We have read this thesis and recommend its acceptance:

Kenneth H. Orvis, Henri D. Grissino-Mayer

Accepted for the Council:
Carolyn R. Hodges

Vice Provost and Dean of the Graduate School

(Original signatures are on file with official student records.)

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and recommend its acceptance:

Kenneth H. Orvis

Henri D. Grissino-Mayer

Acceptance for the Council:

Vice Chancellor and Dean of Graduate Studies

Thesis
2005
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MACROSCOPIC SEDIMENTARY CHARCOAL AS A PROXY FOR PAST FIRE IN NORTHWESTERN COSTA RICA

A Thesis

Presented for the

Master of Science

degree

The University of Tennessee

Kyle James Schlachter

August 2005

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ABSTRACT

Proxy records of fire history offer valuable information on the role fire plays in an ecosystem. Such information can be used to understand the inter-relationships among fire, humans, and the environment. In the past, the forests of Costa Rica were thought to have been spared from significant pre-Columbian forest disturbance; however, paleoecological studies have shown that this notion is false. While the long-term human influence is now recognized in Costa Rica, the nature and extent of human effects in different regions remain poorly documented. Knowledge of pre-Columbian land uses in Costa Rica increases our understanding of the possible impacts of past human disturbances on natural ecological patterns and functioning.

I studied evidence of prehistoric fire provided by fossil charcoal in a sediment core recovered from a lake in northwestern Costa Rica. I developed a new method for the preparation of samples for macroscopic charcoal analysis, and produced an 8000-year-long high-resolution fire record from macroscopic charcoal. My new method of preparing macroscopic charcoal samples involves treatment of sediment samples with 3% U.S.P. cosmetic grade H_2O_2 for 24 hours to deflocculate the sediment and bleach some of the non-charcoal organic matter.

My high-resolution macroscopic charcoal record revealed that fire has been a part of the seasonally dry tropical lowland forest ecosystem in northwestern Costa Rica for the past 8000 years. I found that charcoal influx and fire frequency were greater during the early to middle Holocene and were lower during the late Holocene. After approximately 3600 cal yr BP, macroscopic charcoal influx dramatically decreased. The

timing of this decrease corresponds to archaeological and palynological evidence of human occupation near my study site. Charcoal influx remained low until the arrival of European settlers. I suggest that the increase in charcoal influx at this time is associated with converting forested areas into pasture and the introduction of exotic grass.

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INTRODUCTION

Proxy records of fire history offer valuable information on the role fire plays in an ecosystem. Such information can be used to understand the inter-relationships among fire, humans, and the environment. The principal approaches for developing such records involve dendrochronological study of fire-scarred trees and paleoecological analysis of charcoal fragments recovered from lake sediments. Each of these techniques has advantages and disadvantages.

Dendrochronological techniques have the advantage of accurately dating a fire in time and precisely locating a fire in space. A fire scar, on trees that produce annual growth rings, can be dated to a specific year and often to a specific season of the year using crossdating and intra-ring measurements. Similarly, the presence of a fire scar on a standing tree or standing snag also indisputably places a fire event at the location. However, dendrochronological records often span only several hundred years and at most several thousand years. Conversely, records developed from charcoal particles in lake sediments often extend over many thousands or even tens of thousands of years. Such long records can provide researchers evidence of how fire and the environment interacted during periods not covered by dendrochronological records. However, the temporal resolution available in most sedimentary-charcoal records is less than in dendrochronological records of fire history.

Charcoal analysis of Quaternary lake sediments involves two main approaches: the analysis of microscopic charcoal and the analysis of macroscopic charcoal (Carcaillet et al., 2001b; Whitlock and Larsen, 2001). Microscopic charcoal particles (<125 μm) are

generally counted on pollen slides and provide information on regional as well as local fires (Clark, 1982; Patterson et al., 1987; Mensing et al., 1999; Tinner and Hu, 2003). Macroscopic charcoal particles are concentrated by sieving and identified and counted using a dissecting microscope at low-power magnification. They have been shown to provide a good record of local fires that burned in, or very near to, the drainage basin from which the core was retrieved (Clark and Hussey, 1996; Whitlock and Millspaugh, 1996; Horn et al., 2000; Laird and Campbell, 2000; Millspaugh et al., 2000; Whitlock and Larsen, 2001). Both approaches have become a major part of paleoecological research.

Until the 1990s, almost all paleoenvironmental studies that used charcoal for inferring past fire occurrence were based on the examination of microscopic charcoal in samples prepared for pollen analysis. Whitlock and Larsen (2001) noted three limitations to using such microscopic or "pollen-slide" charcoal in paleoenvironmental research. First, pollen samples are usually spaced several to tens of centimeters apart in a core, thus leaving gaps of unanalyzed sediment. While this sampling strategy may work well for inferring slow changes in vegetation, it can result in missing fire events that are recorded in the sediments not analyzed. Second, charcoal particles can be fractured during pollen processing, which can cause a falsely greater number of microscopic particles. Third, the source area for microscopic charcoal may be very large. A large source area allows a record of regional burning to be developed but may obscure the signal of local fire events.

Macroscopic charcoal (>125 μm) in lake sediment profiles has been shown to provide a good record of local fires (Clark and Royall, 1995; Whitlock and Millspaugh,

1996; Clark et al., 1998; Laird and Campbell, 2000; League and Horn, 2000; Ohlson and Tryterud, 2000; Gardner and Whitlock, 2001; Whitlock and Larsen, 2001; Brunelle and Anderson, 2003; League, 2003; Tinner and Hu, 2003). Sampling a sediment core for macroscopic charcoal analysis at contiguous intervals can reveal changes in the fire record that are missed by wide-interval sampling of microscopic charcoal. Also, macroscopic charcoal analysis often does not entail the same type of mechanical processing of the sediments used during pollen preparation, and may result in less fracturing of charcoal. For these reasons, I used macroscopic charcoal as my proxy for past fire.

This thesis presents a new method for the preparation of macroscopic charcoal particles from lake sediments, and an 8000-year long high-resolution macroscopic charcoal record of fire history from Laguna Estero Blanco in northwestern Costa Rica. This record constitutes the first such record from the neotropical lowlands. The specific questions that guided my research include:

- Is there a technique for preparing sedimentary macroscopic charcoal particles better suited for my study than techniques that currently exist?
- Has fire been a part of this ecosystem for the length of the record?
- Can a change in the fire record be used to identify the time when humans first occupied the area?
- What was the fire record prior to human settlement?
- How did humans affect the fire record?
- Can changes in human population be inferred from changes in the fire record?

- Do changes in the fire record appear to be a result of changes in watershed vegetation?
- Can human disturbances be separated from natural disturbances based on comparisons between the macroscopic charcoal record and pollen data from the same core?
- How do the findings of this research compare to similar findings within Costa Rica and throughout Central America?

This research is significant because of the lack of other proxies that can be used to develop long-term fire records in the tropics. Dendrochronology is widely used in the mid-latitudes for evaluating precisely in time the occurrence of an event or events that affect a tree (Fritts, 1976; Worbes, 1995). However, while much is known about the growth of temperate species, the growth of tropical species is less well studied (Dezzeo et al., 2003), and long-term tropical fire records have not been developed through dendrochronological techniques. However, lake sediments offer the opportunity to develop long-term fire records. The paucity of proxy records of fire history in the neotropics increases the importance of my study.

This thesis is divided into three chapters. In Chapter One, I review literature pertaining to fire-history research using sedimentary charcoal particles. I describe a new method for the preparation of macroscopic charcoal samples from sediment cores in Chapter Two. In Chapter Three, I present a macroscopic charcoal record from Laguna Estero Blanco, Costa Rica. I conclude and summarize my findings following Chapter Three.

CHAPTER 1

INTRODUCTION TO SEDIMENTARY CHARCOAL

Characteristics

Charcoal is carbonized material created from the incomplete combustion of organic matter such as plant tissue (MacDonald et al., 1991; Whitlock and Larsen, 2001). During and after a fire, charcoal can be transported to and deposited in lakes. Charcoal is highly stable as it preserves well and often withstands chemical treatments. Once the charcoal has been extracted from profiles of lake sediment, it can be identified and quantified. Under a dissecting microscope, the macroscopic charcoal particles appear uniformly black with an iridescent sheen, angular, and retain cellular structure (Clark and Hussey, 1996; Brunelle and Anderson, 2003) (Figure 1.1).

Size

The term "macroscopic charcoal" has been used in many paleoecological studies (e.g. Clark, 1988a; MacDonald et al., 1991; Whitlock and Millspaugh, 1996; Clark et al., 1998; Szeicz et al., 1998; Hallett and Walker, 2000; League and Horn, 2000; Carcaillet et al., 2001b; Gardner and Whitlock, 2001; Greenwald and Brubaker, 2001; Long and Whitlock, 2002; Brunelle and Anderson, 2003; Gavin et al., 2003; Huber and Markgraf, 2003; League, 2003). However, the definition of macroscopic charcoal is not consistent. Almost every study defines macroscopic charcoal according to its own terms. Although

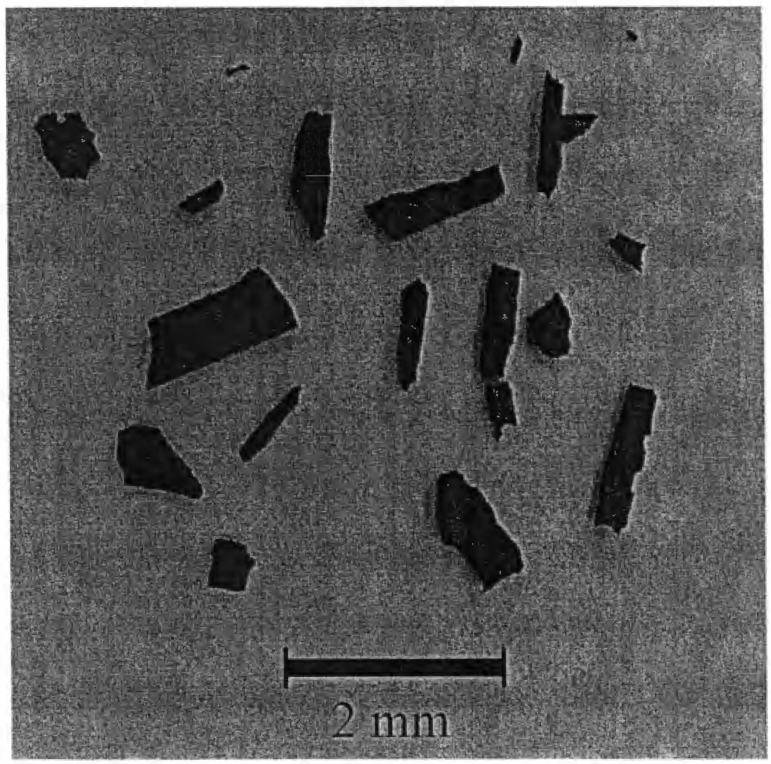


Figure 1.1 Macroscopic charcoal particles after rinsing through a 125-μm sieve.

50 μm has been used as a minimum size, 125 μm is more accepted as a minimum size for macroscopic charcoal (Table 1).

Significance

The choice between using microscopic and macroscopic size ranges in charcoal analysis depends on the signal required to fulfill the research objectives. Macroscopic charcoal analysis is used when the research objective is to understand the local fire record. Yet, the question comes to mind, "How local is local?" Millspaugh and Whitlock (1995) used macroscopic charcoal along with magnetic susceptibility to

Table 1.1 Comparison of minimum particle sizes used in macroscopic charcoal analyses.

Reference	Minimum dimension
Clark, 1988a	50 μm
Macdonald et al., 1991	53 μm
Huber and Markgraf, 2003	63 μm
Whitlock and Millspaugh, 1996	125 μm
Long et al., 1998	125 μm
League and Horn, 2000	125 μm
Millspaugh et al., 2000	125 μm
Brunelle and Whitlock, 2003	125 μm
Szeicz et al., 1998	150 μm
Hallett and Walker, 2000	150 μm
Clark et al., 1998	180 μm
Greenwald and Brubaker, 2001	500 μm
Gavin et al., 2003	500 μm

distinguish local fires, extralocal fires, and regional fires in Yellowstone National Park, USA. They examined the macroscopic charcoal particles in non-laminated sediments and their relationship with known fire events. They studied four small lakes to identify local and extralocal fires and one larger lake to detect regional fire activity. They found that the lakes within recently burned areas contained charcoal in their uppermost sediments and a lake that had no recent fires within its watershed had no macroscopic charcoal in the upper sediments. By comparing the records from each of the five lakes, they concluded that charcoal particles greater than 125 μm in maximum dimension correlated well with fires within a watershed.

Clark et al. (1998) assessed macroscopic charcoal particle transport from an experimental burn in Siberia. They distributed 21 particle traps along three transects extending away from the area of the fire. They counted charred particle areas on each of the traps and then calculated settling velocities for the different particle size categories. Clark et al. (1998) determined that particles 180–250 μm in size have an atmospheric residence time of only minutes and thus concluded that large charcoal particles are an indication of a nearby source.

Gardner and Whitlock (2001) researched the transport and accumulation of macroscopic charcoal (>125 μm) following a fire. They examined cores from 36 lakes within and near the area of a fire in the central Cascade Range of Oregon. They tested whether the fire event was recorded as charcoal peaks in the sediment records of the lakes and examined how the locations of the lakes relative to the burn area affected the presence of macroscopic charcoal within sediment from the lakes. They found that all the cores they examined received charcoal from the fire in question. However, they

noted that lakes within the burned area had better-defined charcoal peaks than sites in unburned areas. They also stated that of the cores examined from unburned areas, sites downwind from the fire had more charcoal than sites upwind from the fire. They concluded that peaks in charcoal >125 μm appear when a fire occurs within the watershed of a lake. The authors suggested that charcoal accumulation from the fire was most strongly related to whether or not the site burned, to the severity of the burn, and to the surface area of the lake.

Ohlson and Tryterud (2000) studied the transport of macroscopic charcoal (>500 μm) from experimental forest fires in Scandinavia. They placed charcoal particle traps at three sites to study the relationship between the deposition of large particles and distance to the fire's edge. They counted and compared charcoal particles from traps inside and outside the burned areas, and found almost no charcoal >500 μm outside the area that was burned. Consequently, they concluded that the presence of charcoal particles >500 μm is a dependable indication of a very local fire event.

These studies demonstrate that the source area for macroscopic charcoal is very local. The significance of macroscopic charcoal as a proxy for fire is that it increases the spatial resolution of the fire record from records developed from pollen slides and other techniques that analyze microscopic charcoal by decreasing the possible source area of the charcoal particles analyzed.

Field Methods

Site selection is an important factor for obtaining the signal of interest in sediment cores (Jacobson and Bradshaw, 1981). As mentioned before, macroscopic charcoal is an

indicator for local fire events. Lakes that are small, deep, and closed make some of the best sites for macroscopic charcoal study (Tolonen, 1986; Millspaugh and Whitlock, 1995; Whitlock et al., 1995). The surface area of a lake is related to the source area of the pollen, charcoal, and other macrofossils that accumulate within the lake. Models suggest that larger lakes accumulate proportionally more material from more distant areas than do smaller lakes (Jacobson and Bradshaw, 1981). Additionally, the depth of the lake is an important factor in the deposition and focusing of charcoal particles. The deepest part of the lake is usually cored because it is where macroscopic charcoal is focused by currents and sediment slumping and where the best record is most likely located (Bradbury, 1996; Whitlock and Millspaugh, 1996). Lakes that lack inflowing and outflowing streams reduce noise that could be caused by fluvial addition of charcoal from distant sources or the loss of charcoal in stream outflow.

In most cases, sediment is taken in the form of a "long" core by using a piston corer, percussion corer, or a vibracorer and then is returned to the laboratory for analysis (Whitlock and Anderson, 2003). Additional "short" cores may be obtained with gravity corers or freeze corers (Clark, 1988b; Whitlock and Millspaugh, 1996). Frozen cores are brought to the laboratory and kept frozen whereas unfrozen short cores are often extruded in the field at 1-cm intervals and stored in plastic bags (Whitlock and Anderson, 2003).

Laboratory Methods

Once a sediment core has been taken from a lake, the researcher must identify and quantify the macroscopic charcoal particles. Several different techniques have been

reported, including sieving, analysis of thin sections, combustion, and automatic imaging. I discuss the methods in detail and consider the advantages and disadvantages of each.

Sieving

The first mention of a sieving method for macroscopic charcoal was made by MacDonald et al. (1991). However, many studies cite Whitlock's research (Millspaugh and Whitlock, 1995; Whitlock and Millspaugh, 1996) as the source for the sieving method (e.g. (Long et al., 1998; Hallett and Walker, 2000; Laird and Campbell, 2000; Millspaugh et al., 2000; Carcaillet et al., 2001b; Brunelle and Anderson, 2003; Huber and Markgraf, 2003; League, 2003). As a result, this method has come to be called "The Oregon Sieving Method" (Laird and Campbell, 2000). This approach is probably the most widely used method to identify and quantify macroscopic charcoal particles.

This method is straightforward. I present the procedure as summarized from Whitlock and Anderson (2003). The equipment and materials needed are: sodium hexametaphosphate, small beakers, spray bottle or a spray attachment for a faucet, metal mesh sieves (sizes most often used are 125 μm, 250 μm, and 500 μm), petri dishes (glass or plastic), and a stereomicroscope. After the core is split lengthwise, subsamples of a known volume of sediment, usually 1.0–5.0 cm^3, are taken contiguously from the center of one half of the core. The required volume of sediment depends on the concentration of charcoal in the sediment. Highly concentrated charcoal particles are easier to identify and count using a smaller volume of sediment. Each subsample is then soaked in a beaker with a dilute solution of sodium hexametaphosphate and water for 24–72 hours.

Sodium hexametaphosphate deflocculates the sediment and separates charcoal particles from clays and other materials.

Once the sediment is disaggregated, the subsamples are gently washed through a series of sieves of different mesh size. After the sieves have been sufficiently rinsed (all particles smaller than the mesh size have passed through) the remaining sediment and charcoal trapped on each sieve is rinsed into a different petri dish.

Charcoal fragments in the petri dishes are identified and counted using a stereomicroscope. Grids etched into the petri dishes or printed on paper placed underneath them help the researcher avoid duplicate counts. A count sheet allows all the particles in one section to be tallied before the next section is counted. After all sections have been counted, the total number of charcoal particles can be tabulated.

The raw charcoal counts obtained using this procedure can be presented as charcoal particle numbers per volume sediment (concentration) or particles/cm^2/yr (influx). A software package is available from the Environmental Change Research Group at the Department of Geography, University of Oregon that can decompose the record into peak and background charcoal signals (Long et al., 1998). Some studies measure the area of the particles (Clark and Royall, 1995; Earle et al., 1996; Carcaillet et al., 2001b) but that technique is more time intensive and may be unnecessary, as studies have revealed total charcoal areas in samples to be highly correlated with fragment numbers.

Advantages to the sieving technique are that it is easy to use, and allows for high-resolution records. Large charcoal particles or other organic macrofossils can be removed and used for AMS-dating (Whitlock and Larsen, 2001; Whitlock and Anderson,

2003). A disadvantage to this technique is that it is time consuming. Rinsing and counting one level can take more than one hour depending on the amount and type of sediment that has to pass through the sieves and the total number of charcoal particles to be counted. Also, the physical process of sieving may fragment larger charcoal particles into two or more smaller particles. Such an alteration could bias counts. One other disadvantage is the visual identification of charcoal particles. Subjective identification of what is charcoal and what it is not could introduce error into the analysis. However, as a long as only one person is counting charcoal and is consistent in his or her identifications, this should not be a problem. The subjectivity of charcoal identification is a bigger problem when comparing separate studies. The inability to see charcoal particles amid obscuring mineral or organic debris is another problem associated with the visual identification of charcoal particles.

Thin Sections

Another common technique for identifying and quantifying macroscopic charcoal uses petrographic thin sections. Thin sections are primarily used with annually-varved sediments (Whitlock and Larsen, 2001; Whitlock and Anderson, 2003). Thin sections are prepared by dehydrating lake sediment, impregnating the sediment with epoxy resin, and then sectioning the sediment (Clark, 1988b). The thin sections are cut into pieces that are small enough to view under a microscope. The charcoal particles are viewed in their original position within the sediment and counted and/or measured.

Advantages to this technique are that the charcoal particles are not altered or fragmented, which is not the case with pollen-slide charcoal and sieving-method charcoal

(Clark, 1988b), and the possibility of annual or sub-decadal resolution if sediments are varved (Whitlock and Anderson, 2003). However, disadvantages to this technique are that varved sediments are rare (Whitlock and Larsen, 2001; Whitlock and Anderson, 2003) and that distinguishing charcoal from unburned plant material can be problematic (Clark, 1988a). The lack of varved-sediment records is reason enough to make the sieving method more practical for a majority of research sites.

Combustion

A less common technique for charcoal quantification is the digestion-combustion method. Winkler (1985) suggested using a nitric acid digestion and a loss of weight on ignition technique as a way to measure the weight of charcoal present within a sediment sample. After using nitric acid to digest all organic material, carbonates, and pyrite in a sample, she determined the amount of charcoal present as a percent of the dry weight of the sample by dividing the difference in dry weights of post-nitric acid treatment and post-combustion by the dry weight of the sample. Her results were comparable to results that relied on optically counting microscopic charcoal. She did, however, note that her method was unable to distinguish carbonized particles from fossil fuel burning from natural charcoal. As a result, charcoal in such sediments would have to be separated microscopically.

An advantage of this technique is that visual identification of charcoal is not necessary. However, the inability to distinguish among carbonized materials and different size categories of charcoal is a problem. Without such distinctions the signal that is obtained from this procedure is indistinct at best.

Automatic Imaging

A problem with visual counting of macroscopic charcoal is the ability of the researcher to accurately distinguish between charcoal particles and other dark matter that may be confused with charcoal, such as pyrite, unburned plant tissue, or insect parts, or to see small particles obscured by fecal pellets or other sedimentary components (League and Horn, 2000). Also, it is possible for different researchers to subjectively identify charcoal differently. A way to circumvent this is to use an automated image analysis system (MacDonald et al., 1991; Horn et al., 1992; Clark and Hussey, 1996; Earle et al., 1996). Image analysis can be used on pollen-slide charcoal (Horn et al., 1992), sieving-method charcoal (Earle et al., 1996), and thin sections (Clark and Hussey, 1996). The general idea for automated image analysis is to use a camera-equipped microscope and computer software to identify charcoal particles. The pollen slide, petri dish, or thin section is scanned and the image is digitized. An algorithm is used that identifies black pixels that surpass a predetermined threshold value as charcoal. Horn et al. (1992) showed that microscopic charcoal counts made with an automated image analyzer and with standard visual methods were very similar.

An advantage to this technique is that it avoids the need for time-consuming visual counting. However, great care must be taken to avoid misidentifying dark material that is not charcoal (Whitlock and Anderson, 2003). Problems with this technique are being worked on presently in the Laboratory of Paleoenvironmental Research at the University of Tennessee.

Prior Studies of Macroscopic Charcoal in Lake Sediments

Whitlock and Millspaugh (1996) tested assumptions about the sedimentation of modern charcoal in lakes of the Yellowstone region. The goal of their research was to empirically examine the spatial and temporal variability of charcoal accumulation in eight lakes in Yellowstone National Park following the 1988 fires. Between 1989 and 1993, they collected 6-cm deep sediment cores along transects across the lakes and used the sieving method to extract charcoal >63 μm. The transects paralleled prevailing wind direction and extended from shallow to deep water. They found that initial charcoal accumulation was greatest in the littoral zone near the downwind shore of the lake. They attributed the littoral zone accumulation to the charcoal being transported there by wind-generated currents. Whitlock and Millspaugh (1996) also found that the charcoal particles were redeposited to the deeper parts of the lakes through time. This redeposition suggested that charcoal remained mobile in this lacustrine system for several years. They concluded that deep areas of lakes are the best locations for collecting sediment to be used in macroscopic charcoal studies because the deepest part of a lake showed a more consistent pattern of accumulation than the shallower areas.

Earle et al. (1996) examined macroscopic charcoal in lake sediments from north-central Alaska. They assessed the relationship between charcoal in sediment-water interface samples and recent fire events at 29 lakes and developed a late-Quaternary record for one lake that spanned the past 14,000 years. They found wide variability in modern sedimentary charcoal in lakes that are in watersheds subjected to similar degrees of burning. They suggested a variety of possible factors that could have caused this variability. These factors are: wind characteristics, unevenness in charcoal deposition

within the lake basin, other fire characteristics, and incorrect identification of charcoal particles. Earle et al. (1996) also suggested that large charcoal particles may be too rare to be good proxies for fire. In addition, they recommended the image analysis method because of its efficiency in counting and measuring large numbers of particles.

Long et al. (1998) outlined a method for decomposing charcoal accumulation rates (CHAR) into background charcoal and peak charcoal elements. The background component consists of charcoal that is mobile within the watershed (trapped in the littoral zone and coming off of the adjacent slopes through overland flow) and regional charcoal that comes from fires in the region but not from within the watershed. The peak component corresponds to charcoal delivered to the lake from a single fire event within the watershed. The background level is determined by calculating weighted averages in a moving window along the CHAR series. The peak events were defined as times when the CHAR series exceeded a predetermined threshold ratio, usually ca. 1.1 times greater than the background component. Long et al. (1998) suggested that breaking down the macroscopic charcoal signal into these two elements aids in evaluating charcoal records.

Long et al. (1998) also developed a 9,000-yr fire record from the Oregon Coast Range that made use of their CHAR method. They experimented with different window-width and threshold ratios to decompose the CHAR series into the background and peak components. They decided that a 600-yr window and a peak threshold of 1.12 were optimal and identified fire events in the watershed for the last 9,000 years. Supporting their background/peak model, they recovered a short core with a ^{210}Pb chronology and identified peaks of charcoal that matched the historical record. Using the calculated peaks for the entire long core, they inferred fire frequency as the number of fire events

per 1,000 years. They found that fires were most frequent in the early Holocene when the climate was warmer and drier. Long et al. (1998) noted that the pollen record (Worona and Whitlock, 1995) also suggested that the fire frequency decreased in the middle and late Holocene when fire-sensitive species became more abundant.

Szeicz et al. (1998) analyzed pollen, macroscopic charcoal (>150 μm), and chrysophycean stomatocysts to create a high-resolution paleoecological record of natural and human-caused disturbances in southern Chile over the past 1,600 years. They employed a variation of the sieving method to identify and quantify charcoal particles from a 58-cm sediment core. Their results suggested evidence of anthropogenic burning following European settlement in the area in the early 1900s as well as evidence that fires were a natural, yet small, part of the system before human occupation. They concluded that high-resolution macroscopic charcoal analysis can help bridge traditional paleoecological and ecological studies.

To extract both a regional and local signal, Laird and Campbell (2000) compared the sieving method and a variation of the combustion method on sediments from a lake in the boreal forest of Alberta, Canada. They used a total carbon analyzer (TCA) for the combustion method rather than loss on ignition. They used the sieving method to provide a local signal and the combustion method to provide a regional signal. The records generated from each method were compared to examine the "spatial sensitivity" of both signals. Surprisingly, their results suggested that both methods provided similar local signals. Both methods yielded records with peaks in charcoal that matched the fire history of the watershed for the last 100 years. Neither method detected large fires within the region but outside the lake's drainage basin. They concluded that the sieving method

was very sensitive to fires along the shore of the lake and that the TCA method detected fires throughout the entire watershed.

Millspaugh et al. (2000) used macroscopic charcoal analysis to develop a 17,000-yr fire record in Yellowstone National Park. They suggested that fire frequency variations were strongly correlated with the July insolation anomaly for the duration of their record. They suggested that regional changes in climate (which are a result of changes in seasonal insolation patterns) were the causes for changing fire frequency as interpreted from charcoal influx using the CHAR model presented by Long et al. (1998).

Carcaillet et al. (2001b) compared charcoal analysis based on pollen-slide microscopic charcoal to macroscopic charcoal based on sieving. They found that both methods showed similar results; pollen-slide charcoal and sieve charcoal were significantly correlated ($r=0.7$, $p<.0001$, $n=266$). They decided that the differences in the two records probably reflected different source areas, and concluded that the sieving method most likely provided a local signal and the pollen-slide method probably provided a regional signal.

Philibert et al. (2003) compared macroscopic charcoal (>150 μm) to the diatom record from Lac à la Pessière in western Quebec, Canada to link fire events with the changes in the diatom record caused by fire-caused changes in phosphorous and CO_2 concentrations in the lake. Unexpectedly, they found that fire events in this coniferous-forest site did not affect limnological characteristics and in turn did not affect the diatom record. They stressed that further tests are needed to confirm their results and that future studies that examine the long-term relationships between aquatic and terrestrial processes are important.

Macroscopic charcoal analysis has also been used in the neotropics. League and Horn (2000) used the sieving method to develop a high-resolution record of fire for the Chirripó páramo in Costa Rica spanning the last 10,000 ^{14}C years. They used macroscopic charcoal analysis to investigate Holocene climate variability and possible human disturbances. They noted that the greater influx of charcoal at the lake during the last 4,200 ^{14}C years could be attributed to drier climate as well as to increased human occupation of the lower slopes of the area, which may have increased ignitions in the páramo by visitors or from fires that spread upslope from agricultural or cooking fires. Although they sieved samples using screens with mesh sizes of 125, 250, and 500 μm, they could only quantify charcoal particles on the 500 μm sieve because of abundant fecal pellets that obscured charcoal on the 125 μm and 250 μm sieves. For those size classes, they recorded only the presence or absence of macroscopic charcoal.

Kennedy (1998) examined pollen, microscopic charcoal, and macroscopic charcoal in Cantarrana Swamp at the La Selva Biological Station in Costa Rica. She determined the presence or absence of macroscopic charcoal (>250 μm) at each of the levels used in her pollen analysis, as a means of reconstructing local fires. She found that local fires were relatively common near the swamp throughout the 3300 cal yr period covered by the core. She noted that charcoal recovered could reflect fires of various origins, but that humans were the likely ignition source given the archaeological and palynological evidence.

Anchukaitis and Horn (2005) reconstructed fire history from Laguna Santa Elena, in southern Pacific Costa Rica, again through coarse interval analysis of sediments from the same levels sampled for pollen and microscopic charcoal analyses. They found that

most of the macroscopic charcoal particles identified were associated with anthropogenic alteration of the landscape at about 1400 cal yr BP near the beginning of their record. The quantity of macroscopic charcoal particles steadily decreased until about 700 cal yr BP, after which point macroscopic charcoal nearly disappeared from the record. Neither Kennedy (1998) nor Anchukaitis and Horn (2005) sampled contiguously to produce a complete fire history.

Macroscopic charcoal is a powerful proxy that provides a record of local fire activity within the watershed of a lake. Much research has been done in the past 15 years that has helped researchers understand the assumptions behind the use of this proxy. The ability to provide a local signal with a high degree of temporal resolution makes macroscopic charcoal analysis a tool that can and should be used in conjunction with other proxies. Current and future research efforts will enlarge the network of paleoenvironmental research sites with high-resolution charcoal records and will contribute to improved interpretations of macroscopic charcoal records.

CHAPTER 2

THE USE OF HYDROGEN PEROXIDE IN PREPARING SAMPLES FOR MACROSCOPIC CHARCOAL ANALYSIS

Introduction

I explored methods for isolating and quantifying macroscopic charcoal in Holocene lake sediments. My reasons for doing so were twofold. First, I sought a method that quickly disaggregated the sediment samples without damaging charcoal particles because Whitlock and Millspaugh's (1996) method of using sodium hexametaphosphate proved insufficient. Second, I wanted to improve visual identification and quantification of charcoal particles by eye or image analysis by making it easier to distinguish charcoal fragments in my samples from dark, non-charred organic matter.

I reviewed various published methods of preparation of charcoal particles from a wide array of disciplines. I evaluated which methods were used and how these approaches affected charcoal particles. My goal was to assimilate the best parts of these different methods into a single procedure that best achieved my needs.

Review of Literature

To determine the effects of different processing techniques on microscopic charcoal particles, Clark (1983; 1984) processed a mixture of fresh wood and grass charcoal in 20 different ways. These treatments included chemical and mechanical approaches: for example, treatments with various acids and oxidants, variations in time of

sonification, centrifugation, stirring, and multiple combinations of these treatments. She found that only vigorous physical treatment increased the total area and number of charcoal particles. She also found that all chemical treatments affected the amount of charcoal but that HCl, HF, and acetolysis did not reduce particle numbers as much as the other treatments. She suggested that analysts avoid the use of strong oxidants, such as Schulze solution, because they had the greatest effect on the charcoal particles, breaking and removing many particles. She also recommended that all samples from a single site be prepared identically to eliminate possible variations arising from the use of different methods.

In a similar comparison study, Winkler (1985) used a nitric acid digestion and a loss of weight on ignition (450–500 °C for 3 hours) technique to measure the weight of charcoal in sediment samples. After using nitric acid to digest all organic material, carbonates, and pyrite in the samples, she determined the amount of charcoal present as a percentage of the dry weight of the samples by dividing the difference between dry weights after nitric acid treatment and after ignition by the dry weight of the samples. Her results were comparable to results that relied on optical counting of microscopic charcoal. She did, however, note that her method could not differentiate carbonized particles caused by fossil fuel burning from natural wood charcoal.

Patterson et al. (1987) described a test of a chemical extraction method carried out by G.L. Jacobson, Jr. (unpublished data). Jacobson tested the combustion method by treating synthetic sediment (composed of known quantities of charcoal and fine sands) with 30% hydrogen peroxide (H_2O_2). This treatment supposedly removed all organic carbon and left only elemental carbon as charcoal. However, the recovery of charcoal

proved to be poor and Jacobson determined that the H_2O_2 must have oxidized some of the charcoal.

Presently, the most common method in use for preparing samples for macroscopic charcoal is the "Oregon Sieving Method" developed by Whitlock and collaborators (Whitlock and Millspaugh, 1996; Long et al., 1998; Millspaugh et al., 2000; Whitlock, 2001; Whitlock and Larsen, 2001), which involves soaking samples in sodium hexametaphosphate prior to sieving. Variations of this wet-sieving technique have also been used (Earle et al., 1996; Laird and Campbell, 2000; League and Horn, 2000; Carcaillet et al., 2001b; Gardner and Whitlock, 2001; Brunelle and Anderson, 2003; Thevenon et al., 2003). Earle et al. (1996) used 10% potassium pyrophosphate instead of sodium hexametaphosphate as a disaggregating agent. Laird and Campbell (2000) used Whitlock and Millspaugh's (1996) method as well as a modified version of Winkler's (1985) method. League and Horn (2000) simply wet-sieved subsamples of 10 cm^3 of sediment through a series of nested sieves. Carcaillet et al. (2001a) deflocculated sediments in a 3% solution of tetra-sodium diphosphate for a minimum of two days and then rinsed the sediments through a metal sieve. Gardner and Whitlock (2001) and Brunelle and Anderson (2003), in addition to the treatment with 5% sodium hexametaphosphate, applied a heated wash of sodium hypochlorite (5% and 10% respectively) to eliminated fecal and other unwanted organic material. Thevenon et al. (2003) used a combination of hydrochloric acid, nitric acid, and H_2O_2. A common denominator in each of these studies, except for League and Horn (2000), is that they all used a chemical treatment to disaggregate the sediments. A need for bleaching or

removing organic material was also reported in several of these studies (Gardner and Whitlock, 2001; Brunelle and Anderson, 2003; Thevenon et al., 2003).

Because my samples contained organic material that needed to be removed, I focused my review of the literature on studies that used chemical treatments to remove organic matter. Rhodes (1998) developed a method for analyzing microscopic charcoal using a 6% H_2O_2 solution. He developed this method to minimize particle fragmentation due to chemical and physical stresses inherent in charcoal preparation procedures and to present a simple, quick, and inexpensive method for charcoal analysis. His method used two 48 hour treatments of 6% H_2O_2 at 50°C. The first stage was designed to initiate the bleaching and digestion of dark organic non-charcoal material. The second stage was used to digest any remaining dark organic matter and to be sure that larger plant fragments were removed. To determine the effect of H_2O_2 on microscopic charcoal, he created fresh charcoal by grinding charred twigs from a biomass fire into known size classes, counted the number of particles in each size class, subjected those particles to his procedure, and then recounted the post-treatment particles. The resulting data indicated that his procedure did not significantly affect the charcoal by fragmentation or digestion.

Many studies used H_2O_2 to prepare soil and sediment analysis samples. White and Hannus (1981) sought a method to estimate charcoal content in soils from grasslands and forests affected by fire and in soils from archaeological sites. They experimented by removing organic and siliceous matter in test samples from soil from an historical forest-fire area in the Black Hills, South Dakota, and from an archaeological site near Belle Fourche, South Dakota. They tested the reactivity of laboratory produced charcoal, prepared by heating branches of *Fraxinus pennsylvanica* in a muffle furnace, with 6%

H_2O_2, 4 M HCl, and 48% HF. Their results showed that charcoal was stable in 6% H_2O_2 at room temperature regardless of particle size or length of the treatment. However, when they tested their procedure on charcoal collected from the archaeological site in South Dakota, they noted that H_2O_2 destroyed uncarbonized organic compounds in the incompletely carbonized charcoal.

Figueiral and Mosbrugger (2000) also recommended the use of H_2O_2 and sodium hexametaphosphate to separate charcoal from soils in archaeological research. Rose et al. (1996) used 30% H_2O_2 to remove unwanted organic matter and carbonates to aid in the identification and enumeration of tephra shards from lake sediments. They noted that this treatment had no effect on the physical appearance of the shards.

Studies that employed particle size analysis techniques frequently used H_2O_2 (Last et al., 1998; Murray, 2002; Allen and Thornley, 2004). These studies noted the ability of H_2O_2 to destroy organic material but made no reference concerning the effect of H_2O_2 on charcoal. K. Orvis (pers. comm.) suggested that H_2O_2 indeed does not seem to destroy charcoal particles that have been subjected to particle size analysis. H_2O_2 is also used in conventional diatom analysis (Battarbee, 1986; Parr et al., 2004) and ostracod cleansing (Curtis et al., 1998) to remove organic material. Drosdoff and Miles (1938) investigated the effect of H_2O_2 on mica during research on soils from the Mojave Desert. They discovered that the use of 30% H_2O_2 destroyed organic matter prior to the mechanical analysis, and exfoliated and altered mica. Smith et al. (1975) used 30% H_2O_2 to remove organic matter without removing elemental carbon as a part of their spectrometric method for determining the amounts of elemental carbon in sediments. Schmidt et al. (1999) evaluated a procedure for dispersing soils for soil texture analysis.

They studied the use of H_2O_2 (10%) for removing organic matter in soils. They found that combusted particles are resistant to oxidation by H_2O_2 at varying degrees. The level of resistance depends on the type and the size of the particles.

The oxidizing ability of H_2O_2 has been adapted for use in a variety of environmental studies encouraging me to test the use of H_2O_2 with a protocol modified from microscopic (Rhodes, 1998) and macroscopic charcoal analyses (Whitlock and Millspaugh, 1996).

Methods

I conducted a series of preliminary experiments to determine the best possible method by which to quantify and identify macroscopic charcoal fragments from the Laguna Estero Blanco sediment core. My tests focused on the different effects of hydrogen peroxide and sodium hexametaphosphate on macroscopic charcoal particles. Hydrogen peroxide disaggregates, removes, and bleaches organic matter, whereas sodium hexametaphosphate only disaggregates sediment samples. I compared the effects of different chemical treatments on duplicate samples, examined the effects of hydrogen peroxide on modern and fossil charcoal particles, and tested the replicability of duplicate counts on the same interval of sediment.

Preliminary Analysis

As a preliminary step, I tested the effects of differing chemical treatments on sediments with differing organic content. I subsampled ten levels from the same duplicate core section from Laguna Los Juncos Near (Haberyan et al., 2003) containing

material deposited between approximately 5640–7570 cal yr BP. Laguna Los Juncos Near is located ca. 1.5 km west of Laguna Estero Blanco. Five of the ten levels sampled were from a more recent interval with higher organic content, and five were from an older interval with lower organic content. The higher organic samples ranged from 40–50% organic on a dry weight basis, and the low from 10–15% organic, based on loss on ignition at 550° C (Dean, 1974). From each level, I took three samples of 1.23 cm^3 and treated one with 5% sodium hexametaphosphate for 24 hours, one with 3% U.S.P. cosmetic grade H_2O_2 for 24 hours, and one with 5% sodium hexametaphosphate for 24 hours followed by treatment with 3% U.S.P. cosmetic grade H_2O_2 for 24 hours. I then rinsed the samples through sieves with mesh sizes of 125, 250, 500, and 1000 μm. The sediment and charcoal remaining on the sieves were dried overnight in petri dishes at 60°C. I counted the particles using a stereozoom microscope at 10–40x magnification. Using Microsoft Excel, I graphed the charcoal concentrations and I calculated the correlation coefficients that existed between charcoal counts of each treatment.

Modern Oak Charcoal

To test the effects of H_2O_2 on charcoal particles, I treated modern oak charcoal samples with different strengths of H_2O_2 (3, 6, and 9% solutions prepared from concentrated reagent grade H_2O_2) and with a control treatment of distilled H_2O. These commercially prepared charcoal samples were provided by K. Orvis, who had already sorted them into size classes by dry sieving (250–500, 500–1000, and 1000–2000 μm). Test samples containing known numbers of particles were prepared for each size range (60 from 250–500 μm, 25 from 500–1000 μm, and 15 from 1000–2000 μm) and were

subjected to treatment in each of the three H_2O_2 solutions for 24 hours. I then rinsed the samples through sieves with mesh sizes of 125, 250, 500, and 1000 μm. The sediment and charcoal remaining on the sieves were dried overnight in petri dishes at 60°C. I counted the particles using a stereozoom microscope at 10–40x magnification. The smallest sieve used in the rinsing served to recover any particles that may have been broken due to chemical or physical stresses. Post-treatment counts were graphed using Microsoft Excel and were statistically analyzed using ANOVA. My null hypothesis stated that no difference existed among pre-treatment and post-treatment counts of the different hydrogen peroxide treatments and the control treatment. The alternative hypothesis stated that the different hydrogen peroxide treatments altered the charcoal particles, and pre-treatment and post-treatment counts therefore differ.

Fossil Charcoal

I next tested the different strength H_2O_2 solutions (1, 3, 6, and 9% solutions prepared from concentrated reagent grade H_2O_2) on fossil charcoal from 24 levels from the same duplicate core section from Laguna Los Juncos containing material deposited between approximately 5640–7570 cal yr BP. This was done to determine if each strength of solution behaved similarly on actual fossil samples. Each different H_2O_2 treatment was applied to six of the samples. From each level, I took two samples of 1.23 cm^3 and treated one with 5% sodium hexametaphosphate and the other with one of the four H_2O_2 treatments for 24 hours. I then rinsed the samples through sieves with mesh sizes of 125, 250, 500, and 1000 μm. The sediment and charcoal remaining on the sieves were dried overnight in petri dishes at 60 °C. I counted the particles using a stereozoom

microscope at 10–40x magnification. I graphed the resulting counts of charcoal particles in Microsoft Excel to determine whether each treatment yielded similar results, and I calculated the counts of H_2O_2-treated charcoal as a percent of counts of sodium hexametaphosphate-treated charcoal.

Modern Grass Charcoal

My results on the previous experiment led me to test the effects of H_2O_2 on modern grass charcoal. I produced charcoal from blades of cane (*Arundinaria gigantean* Walt.) provided by B.E. Wofford, following methods in Orvis et al. (*in review*). I placed dried cane blades in a 25-mL perforated-base porcelain Gooch filtering crucible, surrounded them with 250–500 μm sand, saturated the sand with water, and placed the crucible, for seven minutes, in a furnace heated to 550°C. This method yielded completely charcoalified material that was uniformly black and preserved the cell structure. As with the oak charcoal experiment, I used different strengths of H_2O_2 (in this case 1, 3, and 6%) and distilled water (as a control) on samples with known fragment numbers (10 particles of 250–500 μm size, 10 of 500–1000 μm size, and 5 of 1000–2000 μm size). I broke the charcoalified cane blades and wet sieved the resulting fragments into their respective size classes. I used lower particle numbers in this test than in the oak charcoal tests because of time constraints. Post-treatment counts were graphed using Microsoft Excel and were statistically analyzed using ANOVA. The null hypothesis stated that no difference existed among pre-treatment and post-treatment counts of the different hydrogen peroxide treatments and the control treatment. The alternative

hypothesis stated that the different hydrogen peroxide treatments altered the charcoal particles, and pre-treatment and post-treatment counts therefore differ.

Replication

In my previous experiments, I compared multiple samples, subjected to different treatments, from parallel levels. Because I was uncertain whether horizontally contiguous samples could be meaningfully compared, I tested to see if the results of identical macroscopic charcoal analysis preparation were replicable on identical intervals of sediment. I identified and quantified macroscopic charcoal (125–1000 μm) from 25 levels from the same duplicate core profile from Laguna Los Juncos Near containing material deposited sometime between 1290–3890 cal yr BP (based on dates on overlapping core sections). From each level, I took two samples of 1.00 cm^3 and treated both with 5% sodium hexametaphosphate for 24 hours. I then rinsed the samples through sieves with mesh sizes of 125, 250, 500, and 1000 μm. The sediment and charcoal remaining on the sieves were dried overnight in petri dishes at 60 °C. I counted the particles using a stereozoom microscope at 10–40x magnification. I graphed the resulting charcoal counts in Microsoft Excel to determine whether horizontally contiguous samples treated identically yielded similar results. Using Microsoft Excel, I graphed the charcoal concentrations and I calculated the correlation coefficients that existed between charcoal counts of both treatments.

Results

Preliminary Analysis

The preliminary analyses of treatments on sediments with differing organic contents revealed values that differed, but the general trends are consistent among the treatments (Figure 2.1). Numbers of charcoal particles in the low-organic sediment samples ranged from 53–389 and averaged 223 particles for all treatments. No significant correlation existed between the post-H_2O_2 and post-sodium hexametaphosphate treatments or between post-H_2O_2 and post-sodium hexametaphosphate and H_2O_2 treatments counts of charcoal particles. I found a weakly significant correlation between post-sodium hexametaphosphate treatments and post-sodium hexametaphosphate and H_2O_2 treatments counts of charcoal particles ($r=0.82$, $p<0.10$, $df=3$). Numbers of charcoal particles in the high-organic sediment samples ranged from 483–2593 and averaged 1599 particles for all treatments. No significant correlation existed between the post-H_2O_2 and post-sodium hexametaphosphate and H_2O_2 treatments counts of charcoal particles. I found a weakly significant correlation between post-H_2O_2 and post-sodium hexametaphosphate treatments counts of charcoal particles ($r=0.87$, $p<0.10$, $df=3$) and between post-sodium hexametaphosphate treatments and post-sodium hexametaphosphate and H_2O_2 treatments counts of charcoal particles ($r=0.92$, $p<0.05$, $df=3$).

Modern Oak Charcoal

Counts of modern oak charcoal particles differed slightly (Figure 2.2); however, statistical analysis with ANOVA (Table 2.1) showed that the H_2O_2 post-treatment values

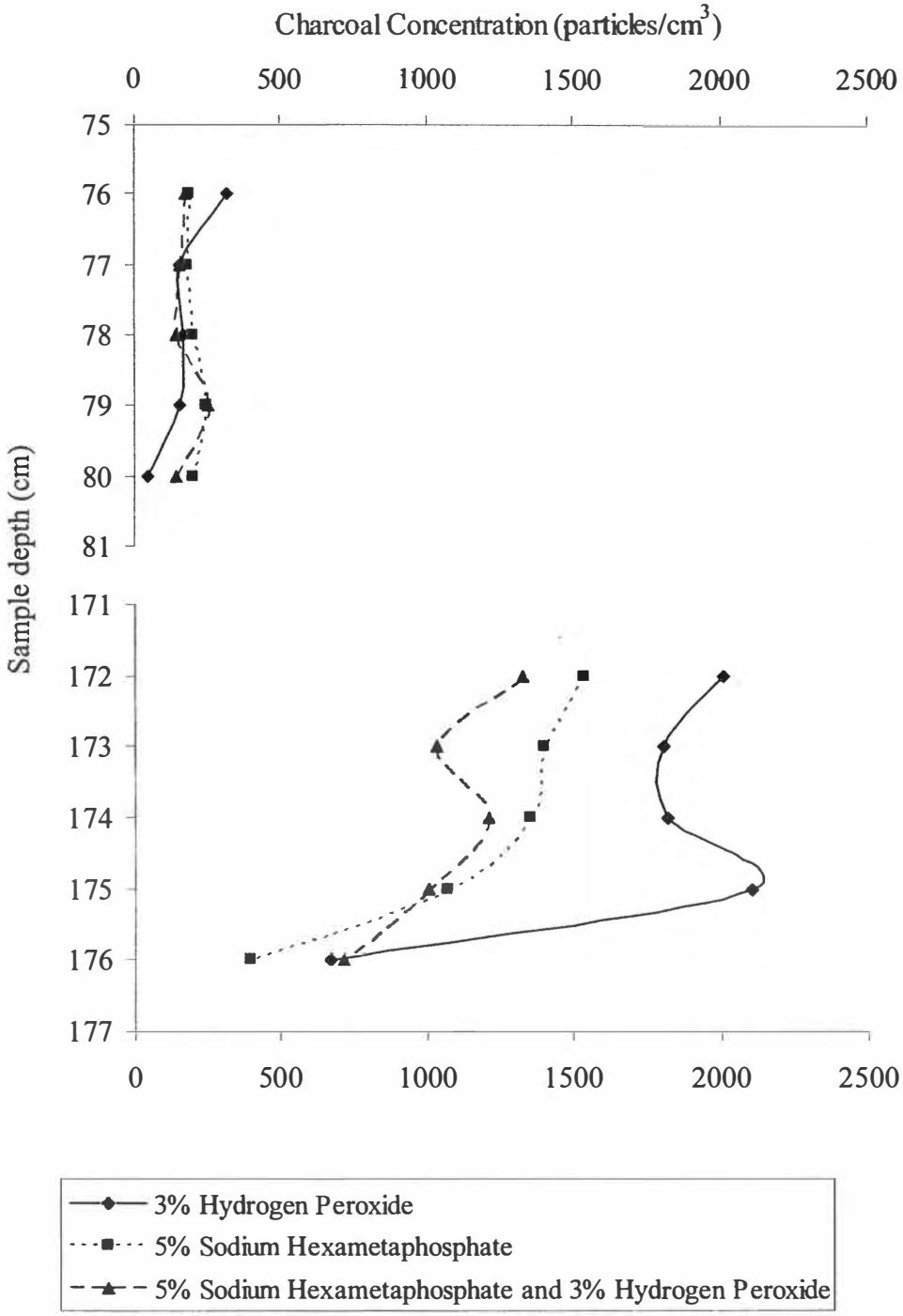


Figure 2.1 Charcoal concentrations in the 125–1000 μm size class from sediments of low (76–80 cm) and high (172–176 cm) organic content at Laguna Los Juncos Near.

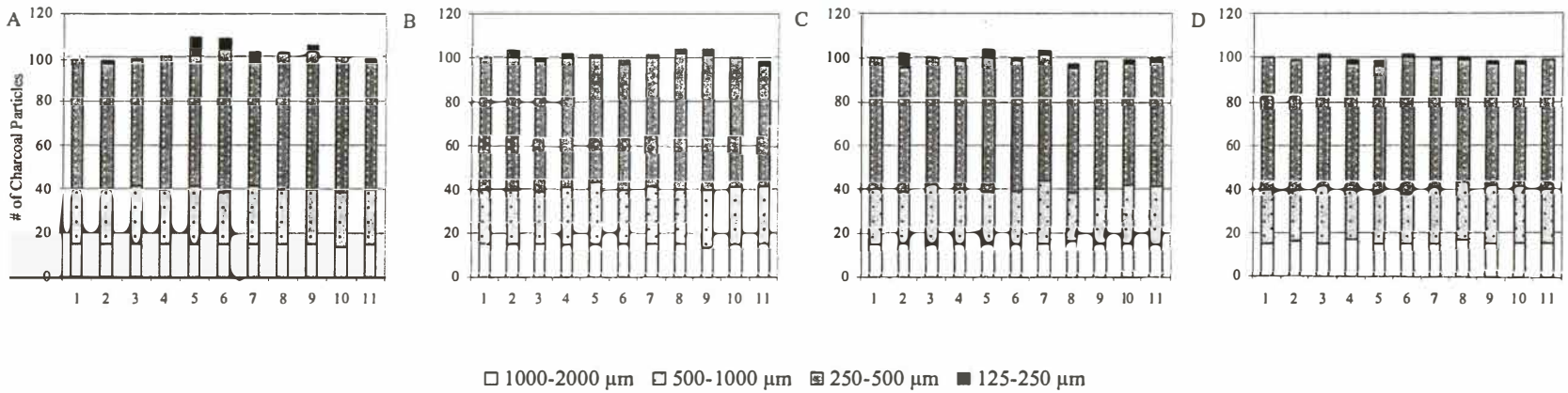


Figure 2.2 Counts of known quantities of modern oak charcoal subjected to different chemical treatments. A) 3%Hydrogren Peroxide; B) 6% Hydrogen Peroxide; C) 9% Hydrogen Peroxide; D) Distilled Water. Sample 1 in each graph is the pre-treatment quantities of charcoal particles.

Table 2.1 Results of ANOVA from counts of modern oak charcoal.

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F-critical*
Between Groups	15.675	3	5.225	1.48	0.235	2.86
Within Groups	126.700	36	3.519			
Total	142.375	39				

of oak charcoal particles were not significantly different from the control (distilled water) post-treatment values.

Fossil Charcoal

Distinct differences exist among the post-treatment counts of fossil charcoal made after different-strength H_2O_2 treatments (Figure 2.3). As H_2O_2 strength increased, the number of charcoal fragments counted decreased (Table 2.2). One percent H_2O_2 and sodium hexametaphosphate yielded the most similar results.

Modern Grass Charcoal

Counts of grass charcoal varied (Figure 2.4), yet statistical analysis with ANOVA (Table 2.3) showed that the H_2O_2 post-treatment values of grass charcoal particles were not significantly different from the control (distilled water) post-treatment values.

Replication

My coincident analyses on horizontally-contiguous samples conducted with the exact same procedure (treatment with 5% sodium hexametaphosphate) show that, while the data from each analysis are not exactly the same, a highly significant correlation exists between the two series ($r=0.50$, $p<0.02$, $df=23$). This statistic indicates that the data show similar trends in macroscopic charcoal signal (Figure 2.5). However, the imperfect match between charcoal values at some levels indicates that fine inferences from minor "wiggles" in charcoal curves are not justified.

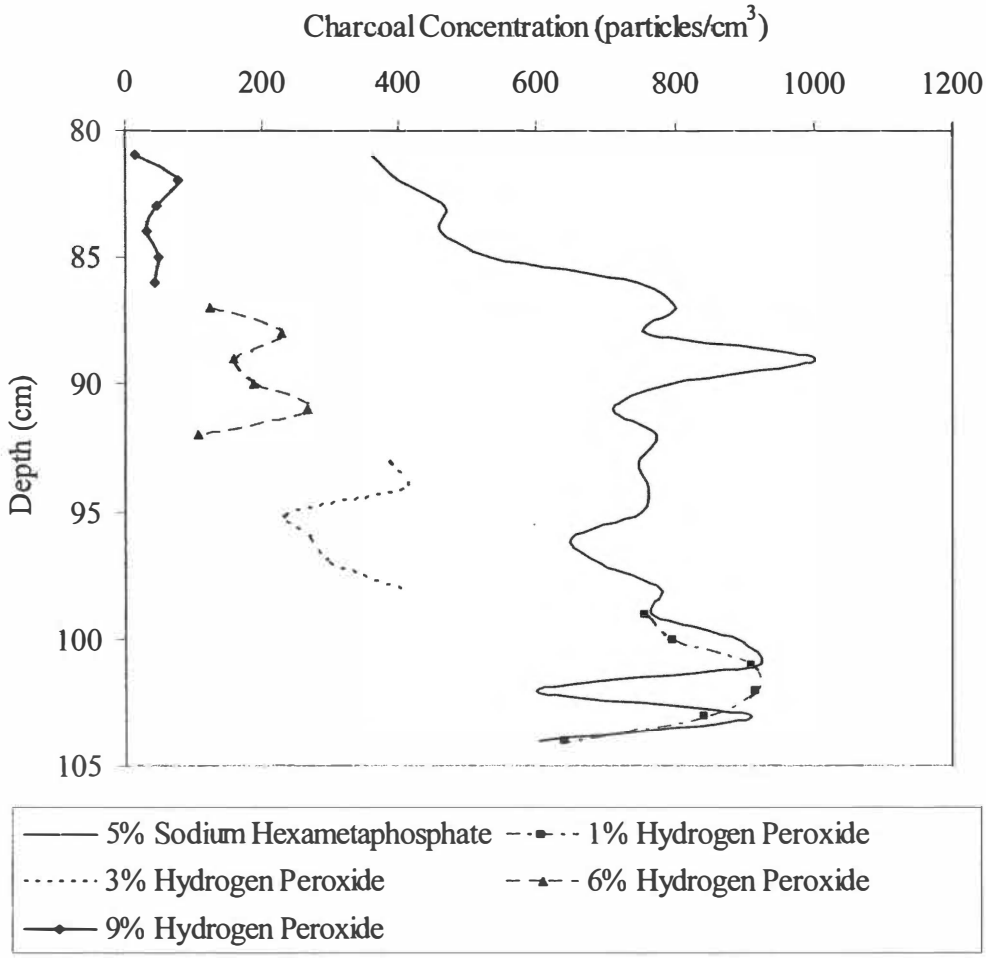


Figure 2.3 Charcoal concentrations in 125–1000 μm size class from sediments from Laguna Los Juncos Near after different chemical treatments.

Table 2.2 Counts of H_2O_2-treated charcoal as a percent of counts of charcoal treated with 5% sodium hexametaphosphate.

Strength H_2O_2	% of $(NaPO_3)_6$ count
9%	9.43
6%	22.83
3%	45.38
1%	106.41

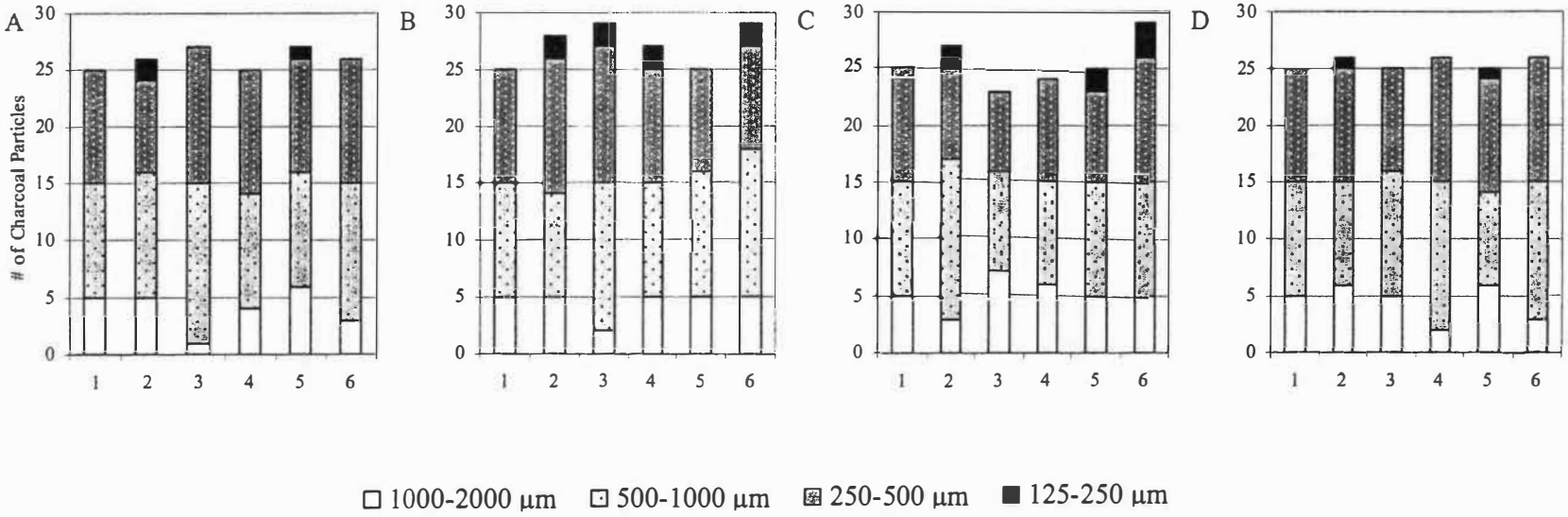


Figure 2.4 Counts of known quantities of modern grass charcoal subjected to different chemical treatments. A) 3%Hydrogren Peroxide; B) 6% Hydrogen Peroxide; C) 9% Hydrogen Peroxide; D) Distilled Water. Sample 1 in each graph shows the pre-treatment quantities of charcoal particles.

Table 2.3 Results of ANOVA from counts of modern grass charcoal.

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F-critical*
Between Groups	13.35	3	4.45	1.85	0.178	3.24
Within Groups	38.4	16	2.4			
Total	51.75	19				

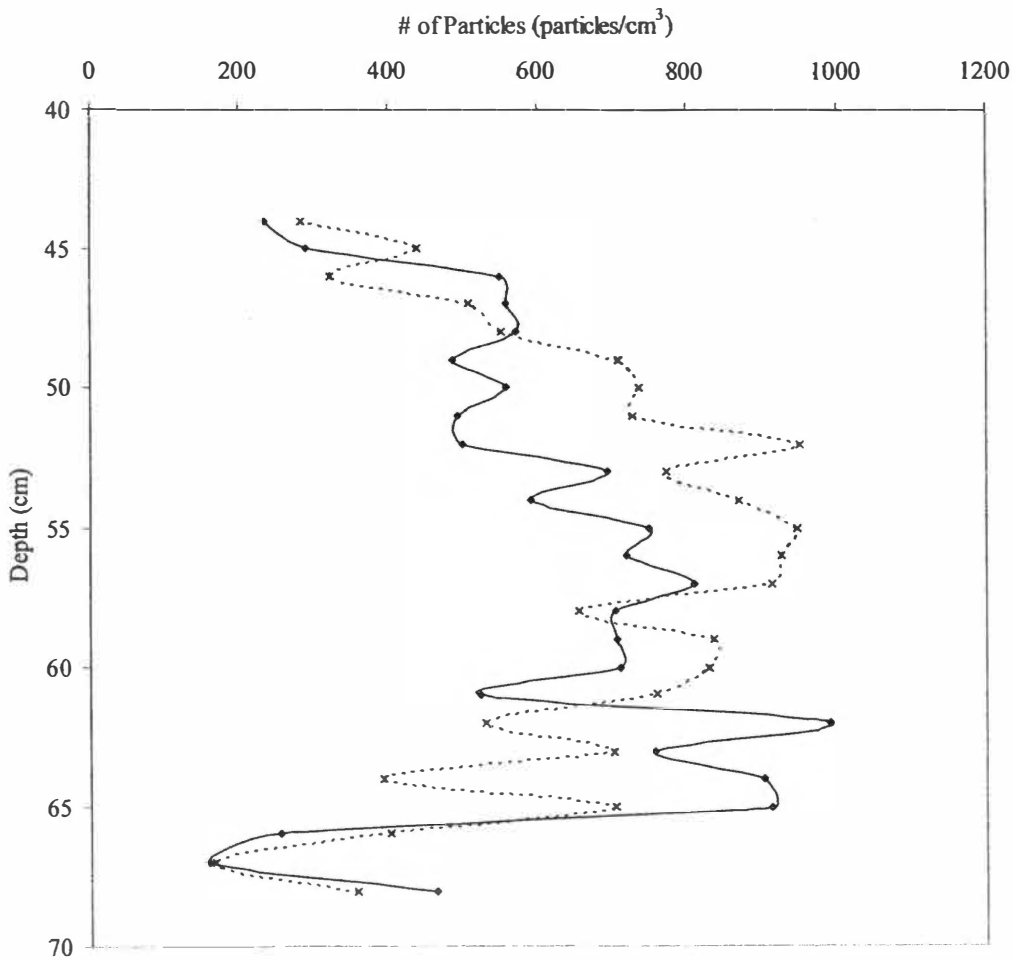


Figure 2.5 Charcoal concentrations in 125–1000 μm size class in duplicate sediment samples from each of 25 levels from the sediment core from Laguna Los Juncos Near, after identical treatments with 5% sodium hexametaphosphate.

Discussion

Preliminary Analysis

Preliminary tests with 3% H_2O_2 (Figure 2.1) were inconclusive and did not definitively prove to me that the different treatments did not affect the charcoal particles differently. That sediments with greater organic content contained more charcoal particles after H_2O_2 treatment than after sodium hexametaphosphate treatment may be due to the need to spend more time rinsing sediments treated with 5% sodium hexametaphosphate through the sieves, owing to greater amounts of organic material that will be left after treatment. This could have led to a greater number of charcoal particles being rinsed through the mesh sieves. It is also possible that duplicate subsamples from the same level within a core may not yield equivalent quantities of charcoal. My additional experiments offered more conclusive evidence about the effect of H_2O_2 on macroscopic charcoal particles.

Modern Oak Charcoal

Statistical analysis with ANOVA showed that the H_2O_2 post-treatment values of oak charcoal particles were not different from the control post-treatment values (Figure 2.2), indicating that completely charcoalified modern hardwood charcoal is unaffected by treatments in H_2O_2 of varying strengths. This suggests that any difference between pre-treatment and post-treatment values can be attributed to mechanical alteration during the rinsing process and/or human error during the quantification process. This resistance to H_2O_2 is consistent with the robust nature of modern hardwood charcoal.

Fossil Charcoal

Comparing the different H_2O_2 treatments performed on sediment core samples to the treatments with 5% sodium hexametaphosphate revealed that fossil charcoal particles are affected differently than modern oak charcoal. Stronger H_2O_2 solutions yield less fossil charcoal than weaker H_2O_2 solutions (Figure 2.3 and Table 2.1). I attribute this discrepancy to charcoal that is not fully carbonized being progressively more digested or bleached by stronger treatments than weaker treatments. These differences in digestion were also found by White and Hannus (1981) and Schmidt et al. (1999). My conclusion is that stronger H_2O_2 solutions more strongly digest or bleach organic matter and thus partially carbonized particles (and other, more spurious dark organic particles) are less stable and are preferentially removed from my charcoal counts.

Modern Grass Charcoal

My experiment with modern grass charcoal indicated that H_2O_2 did not directly alter any completely charcoalified grass charcoal particles. I suggest that any difference between pre-treatment and post-treatment values can be attributed to mechanical alteration during the rinsing process and/or human error during the quantification process.

Replication

The results of my replication experiment indicate that identical sample preparation for macroscopic charcoal analysis yields roughly comparable records of gross fire history. I therefore have confidence that my samples of fossil charcoal treated with different strength H_2O_2 treatments and sodium hexametaphosphate contained roughly

similar numbers of charcoal particles prior to any treatment. Therefore, the major differences between post-treatment counts (Figure 2.3) can be attributed to effects of the treatments. Minor differences such as those seen in my preliminary analyses (Figure 2.1) can be attributed to between-sample differences. I discuss the implications of these replicability limits in my conclusions following Chapter 3.

Conclusions

I conclude that treating samples of known volume of sediment with a known volume of 3% U.S.P. cosmetic grade H_2O_2 for a period of 24 hours is best suited for macroscopic charcoal analysis of the Laguna Estero Blanco sediment core. I believe that using cosmetic grade H_2O_2 (sold in pharmacies in 0.5 l bottles for less than $1 USD) is superior to using diluted concentrated reagent grade H_2O_2 in two ways. First, cosmetic grade H_2O_2 is much less expensive than reagent grade H_2O_2. Second, it contains stabilizers that allow the researcher to be confident that a newly opened bottle will be of the appropriate strength (by law, it must be between 2.5–3.5%). Because H_2O_2 readily breaks down when in contact with air and light, stock bottles of concentrated H_2O_2 used to prepare solutions may be significantly less concentrated at the time solutions are mixed than indicated by the bottle label. Moreover, dilutions, once mixed, are also likely to break down quickly. My suggestion is to open a small and fresh bottle of stabilized cosmetic grade hydrogen peroxide for each batch of samples processed. "Leftover" H_2O_2 can be used for other purposes in the lab or given away for home uses.

The results from these methods should be comparable to those obtained using the "Oregon Sieving Method" (Whitlock and Millspaugh, 1996; Laird and Campbell, 2000).

Using H_2O_2, rather than 5% sodium hexametaphosphate, improved sediment disaggregation in my samples and may be helpful to others working with similar sediments. Some of my samples contained clumps of sediment after treatment with sodium hexametaphosphate whereas no samples treated with H_2O_2 retained sediment clumps. The use of hydrogen peroxide also facilitates the identification and quantification of charcoal particles in organic-rich samples because dark organics that might be confused with charcoal are either bleached or digested (Earle et al., 1996) (Figure 2.6). This method will also yield samples that can be more easily quantified with image analysis because of the removal and bleaching of non-charred organic matter.

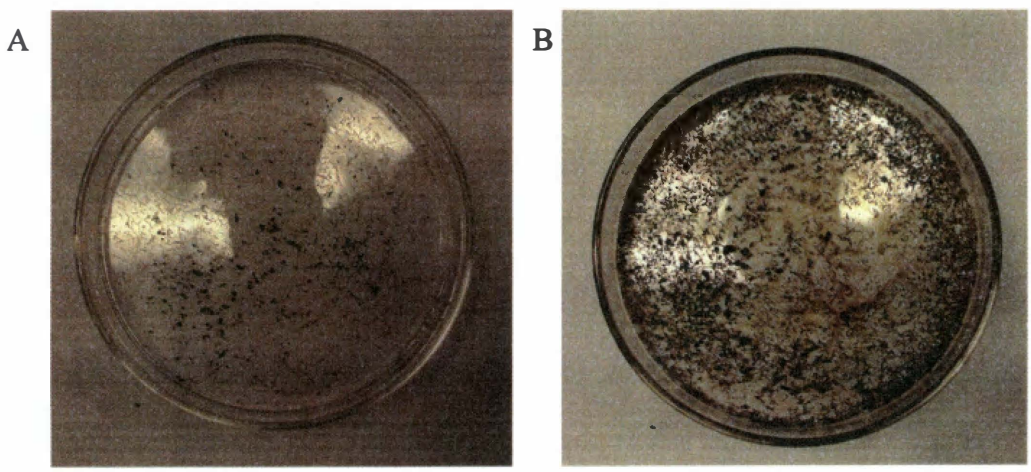


Figure 2.6 Photographs of samples after treated with A) 1% H_2O_2 and B) 5% sodium hexametaphosphate.

The results of my experiments also raised questions about the replicability of macroscopic charcoal analysis. While large-magnitude variability is replicable in neighboring samples treated identically, a considerable amount of small-magnitude variability exists (Figure 2.5). This variability suggests to me that minor wriggles in macroscopic charcoal records using sample sizes of 1 cm^3 may be prone to cross the margin of robust inference in the identification of individual fire peaks. I suggest that further research is necessary to more fully understand this important issue.

CHAPTER 3

AN 8000-YEAR HIGH-RESOLUTION SEDIMENTARY CHARCOAL RECORD FROM LAGUNA ESTERO BLANCO, COSTA RICA

Introduction

In the past, the forests of Costa Rica were thought to have been spared from significant pre-Columbian forest disturbance (Sanford and Horn, 2000). However, paleoecological studies have shown that this notion is false (Horn and Sanford, 1992; Northrop and Horn, 1996; Kennedy and Horn, 1997; Kennedy, 1998; Sanford and Horn, 2000; Clement and Horn, 2001; Horn and Kennedy, 2001; Anchukaitis, 2002; Anchukaitis and Horn, 2005). While the long-term human influence is now recognized in Costa Rica, the nature and extent of human effects in different regions remain poorly documented. Knowledge of pre-Columbian land uses in Costa Rica increases our understanding of the possible impacts of past human disturbances on natural ecological patterns and functioning (Sanford and Horn, 2000). In this chapter, I present evidence of prehistoric fire provided by fossil charcoal in a sediment core recovered from a lake in northwestern Costa Rica. This record constitutes the first such record from the neotropical lowlands.

Research Setting

Environmental Setting

This research involved analyses of sediment cores from six lakes on the lower southwestern slope of Volcán Miravalles, in the Cordillera de Guanacaste in northwestern Costa Rica (Figure 3.1). The Cordillera de Guanacaste is a series of stratovolcanoes that trend NW–SE. Laguna Estero Blanco (10.667° N, 85.204° W) is located at about 430 m elevation just south of the town of La Fortuna de Bagaces. The other lakes in the larger study are: Laguna San Pablo, Laguna Los Juncos Near, Laguna Las Brisas, (all shown in Figure 3.2) Laguna Martínez (shown in Figure 3.2 but not labeled as Laguna Martínez), and Laguna Sorpresa (not shown in Figure 3.2).

Laguna Estero Blanco is a small lake, about 1.5 ha in area with a maximum depth of 2.8 m (Haberyan et al., 2003). The lake occupies a depression formed by lava and lahar flows and by the deposition of other volcanic material (Alvarado, 2000; Haberyan et al., 2003). The area of the watershed occupied by the lake is approximately 10–15 hectares. Limnological studies at Laguna Estero Blanco revealed that the thermocline was located 1.5 m below the surface and that the lake was characterized by high Ca^{+2} content relative to the other ions examined, followed by Si, Na^{+}, and Mg^{+2}, and with K^{+} and Cl^{-} having the lowest concentration (Haberyan et al., 2003). Basal radiocarbon dates indicate that Laguna Estero Blanco and other lakes in the vicinity were formed about 8000 years ago in association with lava and lahar flows from Volcán Miravalles (Alvarado, 2000; Arford and Horn, 2004).

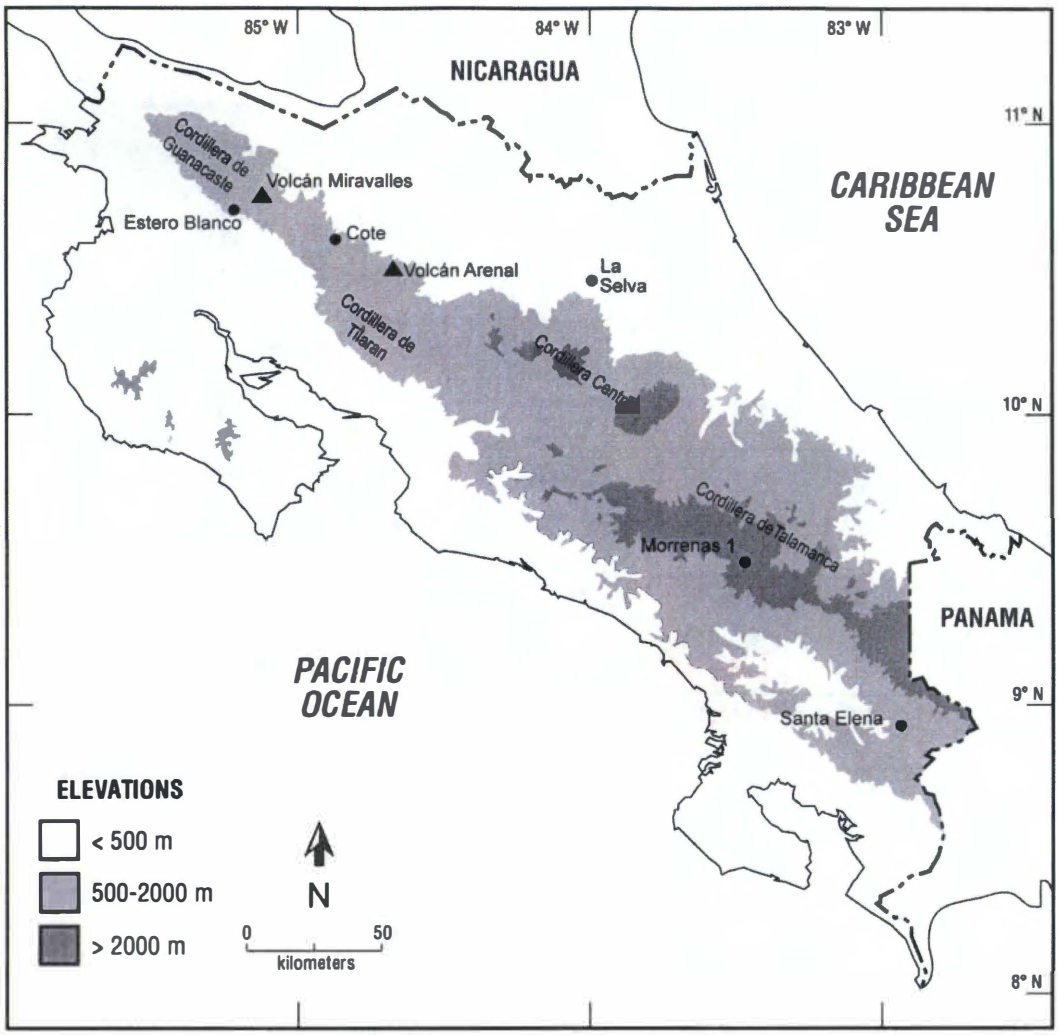


Figure 3.1 Map showing location of Laguna Estero Blanco in Costa Rica and physical features and paleoecological sites mentioned in text. Map modified from Horn (*in review*).

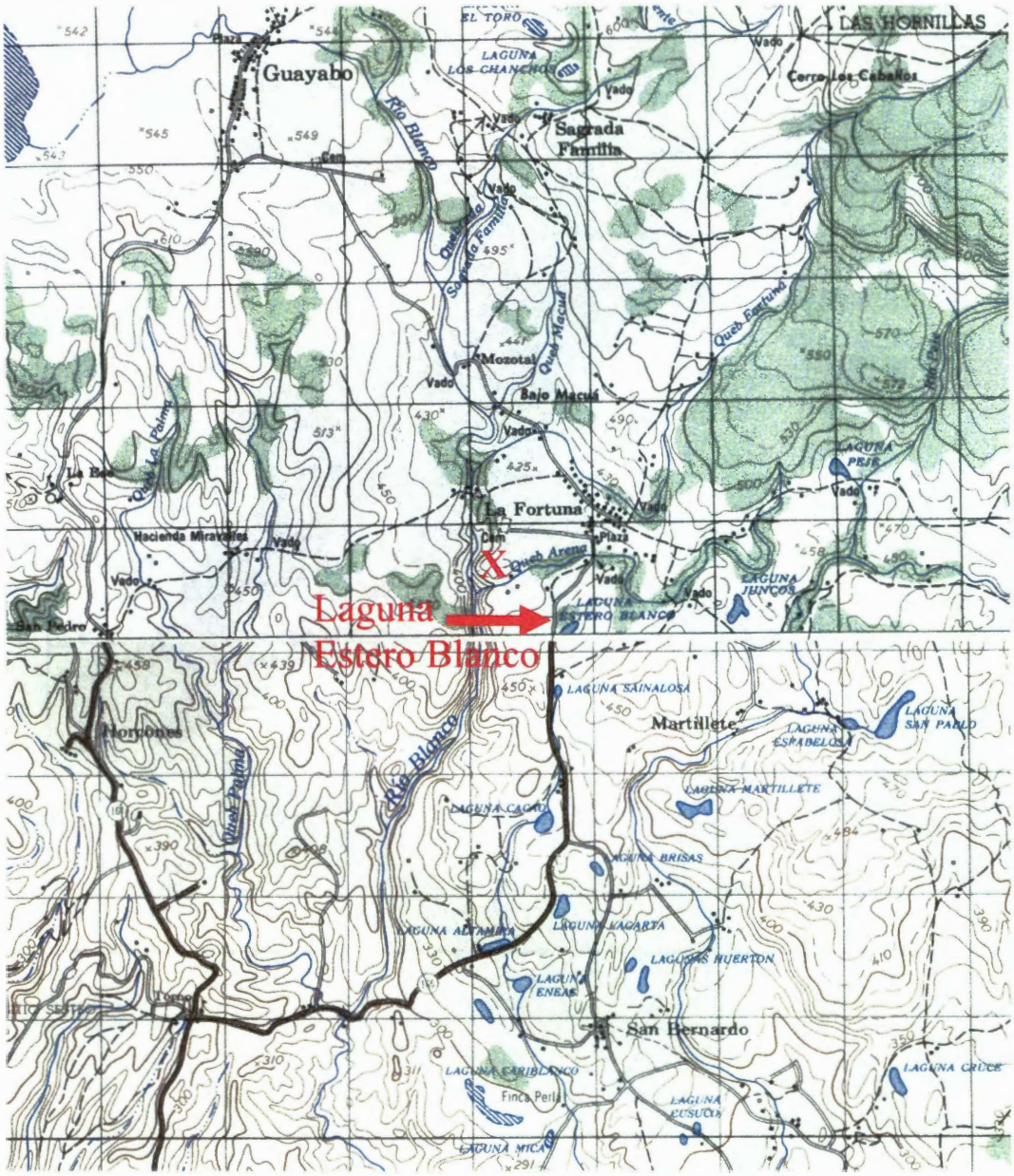


Figure 3.2 Location of Estero Blanco and other lakes in the Miravalles region, as shown on 1:50,000-scale Miravalles and Tierras Morrenas topographic quadrangles. X is location of archaeological site 3148II-9 (Ryder, 1982–1983). For most of the year, Los Juncos is two separate lakes, Los Juncos Near (southwest lobe as mapped) and Los Juncos Far (eastern lobes) (Haberyan et al., 2003). Map adapted from and courtesy of M. Arford.

The lake site is located in the Premontane Moist Forest Basal Belt Transition Life Zone in the Holdridge bioclimatic classification (Bolaños and Watson, 1993). The Life Zone model gives preference to total quantity of precipitation over the seasonal distribution of precipitation (Holdridge et al., 1971). This classification is the same as much of Guanacaste province and half or more of the Area de Conservación Guanacaste in which the management emphasis is on restoring "tropical dry forest" (Allen, 2001).

In the Herrera (1985) climate classification of Costa Rica, the lake site is located within climate group B4, the Subhumid-humid climate, warm with a very long dry season (>70 intermittent days with a water deficit). This climate classification system expresses the idea of seasonality better than the Holdridge Life Zone system. The climate is characterized by a distinct dry season from November to April (Coen, 1983). The mean annual rainfall on the lower slopes of the Cordillera de Guanacaste is about 1500–2000 mm, and the mean annual temperatures in the area range between 22.5 and 25.0 °C (Coen, 1983; Bergoeing, 1998). The lake is surrounded by cattle pasture and scattered clumps of deciduous trees. Gómez (1986) classified the vegetation of this area of Costa Rica as semi-deciduous lowland forest. Throughout the Guanacaste province, these seasonal lowland forests are referred to as tropical dry forests (Allen, 2001).

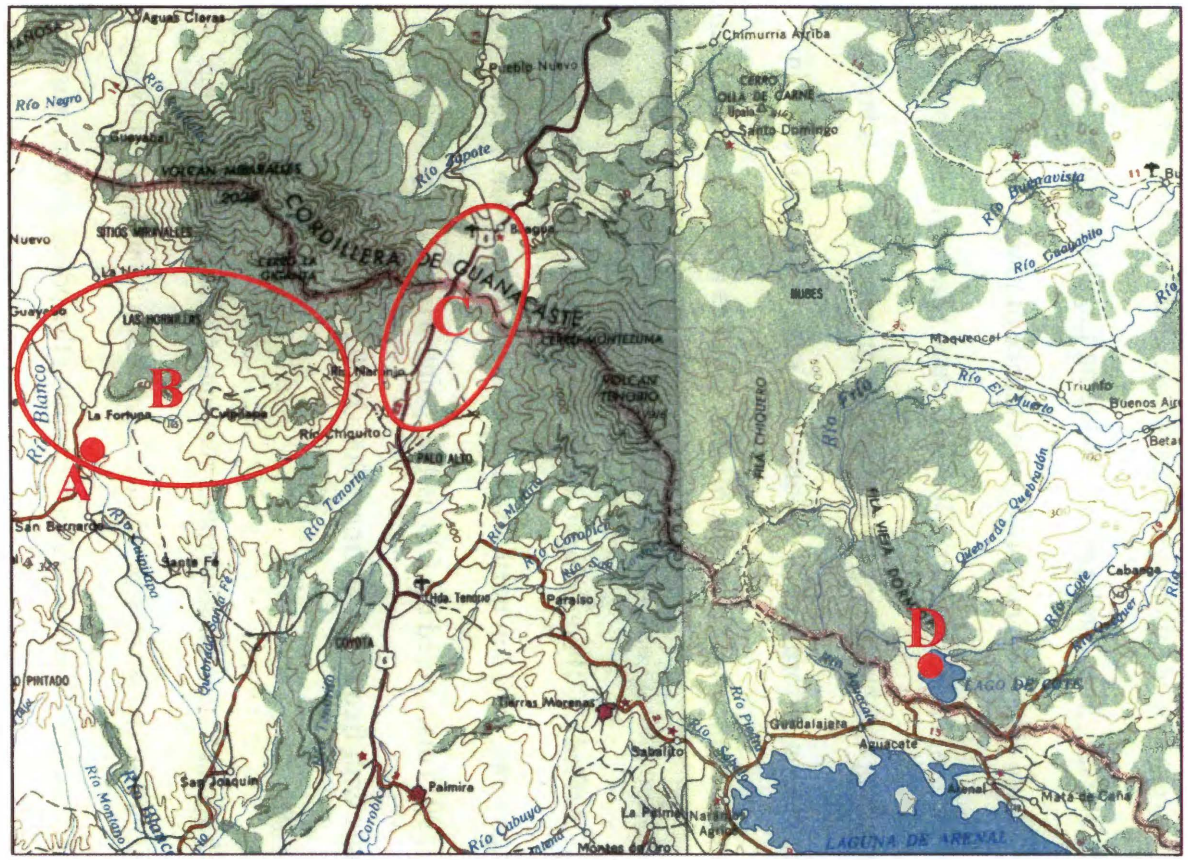
Archaeological Setting

The timing of the peopling of Costa Rica has been under debate because of the paucity of artifacts that provide evidence of early human inhabitants. Evidence of early human occupation of South America as far back as 11,000 ^{14}C yr BP (Bryan and Gruhn, 2003; Gruhn, 2004) suggests that humans must have migrated through Central America,

and necessarily Costa Rica, from North America at some earlier time (Bush et al., 1992). Snarskis (1979) proposed that an undated lithic complex discovered in the Turrialba Valley, in eastern Costa Rica, is evidence of PaleoIndian occupation in Costa Rica. A Clovis-like point was found along the shore of Lago Arenal near Volcán Arenal (Figure 3.1), along the eastern border of the Guanacaste Province (Sheets et al., 1991). The authors suggested that this artifact may indicate the oldest human occupation of the area, possibly dating to 10,000 B.C.

Within the general area of Laguna Estero Blanco, two archaeological studies have been conducted (Norr, 1982–1983; Ryder, 1982–1983) (Figure 3.3). Norr (1982–1983) surveyed and excavated pre-Columbian burial mounds in the Río Naranjo–Bijuagua valley east of Volcán Miravalles. The earliest radiocarbon date she reported for occupation of the study area was 3500 ± 60 ^{14}C yr BP. She concluded that the settlement occurred earlier in the southern part of the valley, the closest part of her study area to Laguna Estero Blanco.

Ryder (1982–1983) surveyed an area near Guayabo de Bagaces that included Laguna Estero Blanco. He surveyed 23 sites of pre-Colombian activity, including 17 cemeteries, 2 habitation sites, 2 habitation plus cemetery sites, and 2 petroglyph sites. He dated these sites to 800 B.C.–A.D. 500. These sites were located north and upslope of Laguna Estero Blanco. The nearest archaeological site (3148II-9) to Laguna Estero Blanco was on a plateau north of Quebrada Arena (Figure 3.2). Scattered sherds were found at this site. Ryder (1982–1983) suggested that the deposits may indicate a habitation site.


A
B
C
D
Río Negro
Aguas Claras
Pueblo Nuevo
Chimurria Arriba
Santo Domingo
Río Zapote
Río Bijuavista
Río Caoabito
CORDILLERA DE GUANACASTE
VOLCAN MIRAVALLES
SITIOS MIRAVALLES
LAS HORMIGAS
La Fortuna
Río Chiquito
PALO ALTO
Río Tenorio
San Bernardo
Santa Fe
Río Blanco
Río Montado
San Joaquín
PINTADO
Palmira
Río Quebuyo
Montes de Oro
Tierras Morenas
Sabalito
Río Piedras
Río Frío
Río Muerto
Maqueacet
Triunfo
Buenos Aires
Quebrada Quebradón
Río Cote
Cabanga
LAGO DE COTE
Guadalajara
Aguacate
Alonal
Mata de Caña
LAGUNA DE ARENAL
Paraíso
Río Tempisque
Río Chiquito
Río Aguacate
Río Piedras
Río Sabalito
Río Tenorio
Río Muerto
Río Cote
Río Quebuyo
Río Montado
Río Blanco
Río Caoabito
Río Bijuavista
Río Zapote
Río Negro
Río Frío
Río Muerto
Río Cote
Río Quebuyo
Río Tempisque
Río Tenorio
Río Chiquito
Río Aguacate
Río Piedras
Río Sabalito
Río Caoabito
Río Bijuavista
Río Zapote
Río Negro
Río Blanco
Río Montado

Together, these two studies provided archaeological evidence of human occupation within the surroundings of Laguna Estero Blanco for the past 3,500 years.

Previous and Ongoing Studies at and Near the Lake

Arford (in progress) developed pollen and microscopic charcoal records for the six lakes that are a part of the larger research project, including Laguna Estero Blanco. His pollen record from Laguna Estero Blanco provides evidence of maize cultivation within the watershed for the past ca. 3300 years. Because maize (*Zea mays* subsp. *mays*) is a plant that requires planting and harvesting by humans, its presence indicates human occupation. Few data other than Arford's exist on long-term environmental history in this region. The closest paleoecological site to the Miravalles lakes is Lago Cote, located ca. 35 km from Laguna Estero Blanco on the southeastern end of the Cordillera de Guanacaste (Figure 3.3). Arford (2001) developed a 4000-yr pollen and microscopic charcoal record from Lago Cote, ca. 3 km north of Lago Arenal and ca. 9 km north of the site where Sheets et al. (1991) discovered the Clovis-like point. His record provided evidence of a drier climate with frequent fires from 4000–2600 cal yr BP and less fire activity after 2600 cal yr BP.

I chose to study Laguna Estero Blanco, rather than one of the other Miravalles lakes, for several reasons. First, the small size of the lake makes it ideal for developing a local fire record from macroscopic charcoal. After examining the pollen and microscopic charcoal records developed by Arford, I learned that Laguna Estero Blanco could be more useful for macroscopic charcoal analysis than the other lakes because of the length of the core, the total number of levels analyzed for pollen, and the marked changes

present in the Estero Blanco pollen record. Additionally, a petroglyph near the shore of Laguna Estero Blanco provides unequivocal evidence of human presence in the immediate surrounding area.

The fire management of seasonally dry tropical forests in Costa Rica has been the topic of considerable research (Murphy and Lugo, 1986; Janzen, 1988; Tenenbaum, 1994; Janzen, 2002; Stern et al., 2002; Kauffman et al., 2003). While the natural occurrence of fire has been documented (Middleton et al., 1997), the popular belief is that fire is not a natural part of these forests (Murphy and Lugo, 1986; Janzen, 1988; Tenenbaum, 1994; Allen, 2001; Janzen, 2002). This paradigm has played a major role in the implementation of the Guanacaste National Park Project, a campaign to restore the forest after centuries of deforestation by farming, ranching, and logging (Allen, 2001). The principal restoration strategy in this project has been to prevent fires in Guanacaste forests. The Area de Conservación Guanacaste Program for Prevention and Control of Forest Fires developed three broad objectives: (1) to reduce the ignition and propagation of forest fires, (2) to provide instruction to staff on how to combat fires, and (3) to raise awareness of the fire problem as a way to push toward more intelligent use of fire (Allen, 2001). However, the past fire regimes of this ecosystem are not well understood. Because of the dynamics of this system, especially relating to human influence, these interactions must be examined at a variety of temporal scales (Kauffman et al., 1993). This study will provide a prehistoric record of fire activity in seasonally dry tropical forests that will add to our understanding of the long-term role of fire in this ecosystem, and will provide information relevant to conservation and restoration of these forests.

Field Methods

Horn, Arford, and others visited the Laguna Estero Blanco site several times between 1998 and 2001 and collected a sediment core from the center of the lake in March 2001. They used a plastic tube fitted with a rubber piston to retrieve the near-surface sediments and a Colinvaux-Vohnout locking piston corer (5-cm diameter; (Colinvaux et al., 1999) to recover deeper sediment in sections of 1 m in length. The near-surface sediments were extruded in the field in 2-cm intervals and stored in plastic bags. The deeper core segments were returned to the Laboratory of Paleoenvironmental Research at the University of Tennessee encased in their original aluminum coring tubes. Core sections were separated by gaps of 5–20 cm, owing to incomplete recovery and the use of a core catcher that resulted in shorter cores.

I participated in fieldwork in the Miravalles study area in June of 2003 and visited Laguna Estero Blanco as well as all of the other lakes included in the larger study. I assessed the topography and the vegetation of the region. This field experience provided me with important insights on how to conduct and interpret my analyses to better understand the fire-related processes that have occurred on the landscape.

Laboratory Methods

Core Opening and Description

The aluminum coring tubes were opened in the Laboratory of Paleoenvironmental Research at the University of Tennessee by S. Horn, M. Arford, and laboratory assistants by cutting the tubes longitudinally with a modified table router. The sediment inside the tubes was then sliced longitudinally using a thin wire. The core sections were

immediately photographed by S. Horn and the stratigraphy of each section was described by M. Arford. Core halves were labeled A and B. Both halves were then refrigerated at 5°C. M. Arford sampled the B core half for pollen and microscopic charcoal analyses.

Chronology

Organic macrofossils from the Laguna Estero Blanco core were selected and removed for radiocarbon dating by M. Arford and S. Horn. The materials were rinsed with distilled water, oven dried, and submitted to Beta Analytic Laboratory and the University of Arizona AMS laboratory for accelerator mass-spectrometry (AMS) dating. I used the CALIB v4.4.2 program (Stuiver and Reimer, 1993) and the INTCAL98 dataset (Stuiver et al., 1998) to calibrate the dates. I created an age-depth model using the weighted mean of the probability distribution of the calibrated radiocarbon dates (Telford et al., 2004). I estimated the ages of the sediment intervals sampled by linearly interpolating between the calibrated dates. Sedimentation rates were assumed to be constant over each interval.

Macroscopic Charcoal Analysis

I removed subsamples of 2.0 cm^3 from contiguous 1-cm intervals in the A half of each core section using a small rectangular brass sampler fabricated by R. Horn from a design I developed with S. Horn (Figure 3.4). Sampling the core material in this manner leaves a groove 1 cm deep and 2 cm wide for the length of the core while contiguously sampling the sediment. Sample preparation followed the method outlined in Chapter 2. Each subsample was treated with 3% U.S.P. cosmetic grade H_2O_2 for 24 hours. The

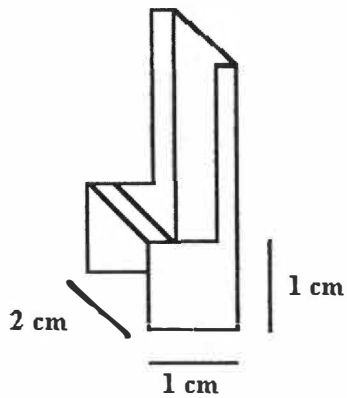


Figure 3.4 2.0 cm^3 brass sampling device.

samples were then gently rinsed through a series of sieves (250, 500, and 1000 μm) using a spray of distilled water from a RL Flo-Master 7.6-l-capacity lawn and garden sprayer. All charcoal particles greater than 250 μm were rinsed into glass petri dishes with distilled water and oven dried overnight at 60°C. Charcoal fragments were tallied using a stereo binocular microscope at 10–40x magnification. Particles that appeared uniformly black with an iridescent sheen, were angular, and showed cellular structure were identified as charcoal.

Data Analysis

Charcoal counts were converted into charcoal concentrations (particles/cm^3). The charcoal concentration data were then divided by the time in years represented by the sample (yr/cm), as estimated by the age-depth model, to calculate charcoal influx (also referred to as charcoal accumulation rates or CHAR) (particles/cm^2/yr). Using a Fortran program written by P. Bartlein (Long, 1996; Long et al. 1998) the charcoal record was

converted into pseudo-annual values and presented in 10-year averaged intervals. Using another Fortran program written by Bartlein (Long, 1996; Long et al., 1998), the CHAR data were divided into background and peak values. The background values were defined as the locally weighted mean accumulation rate. Peak values were defined as an individual accumulation rate exceeding the background value by a predetermined threshold ratio. The background-window width was determined by comparing several different window widths (300, 450, 600, and 900 years). The peak-threshold ratio was determined by examining several different ratios (1.05, 1.1, and 1.15) with each of the different background-window widths. The two programs I used are together known as the Charcoal Analysis Programs or CHAPS.

I had initially analyzed the record as one continuous record, but the presence of a 20-cm gap in the record between the first and second core sections resulted in distortion of the values obtained using the second of the two programs above. Based on advice from C. Long (pers. comm. Feb 2005), I decided to divide the charcoal data into two sections and analyze each separately. I then recombined the resulting data to form one single record. The results of this approach improved my ability to accurately analyze the macroscopic charcoal record. Small sediment gaps in the lower of the two sections remained in the data during analysis with CHAPS, but were so small that they did not affect the output from the programs.

Results

Sediments and Stratigraphy

Coring operations at Laguna Estero Blanco yielded 0.41 m of near-surface sediment (collected in the plastic tube) and 3.95 m of deeper sediment (collected in aluminum core tubes). Correcting for overlap between the sediment core sections, a total of 4.47 m of sediment was cored, and 4.15 m was recovered. This difference could be due in part to compaction, but more often seems to have resulted from incomplete recovery due to material being "plowed" through rather than collected, or because material collected fell out of the bottom of the tubes before recovery was completed. This was not generally a problem because of the use of a core catcher device designed by M. Arford to prevent core loss. Unfortunately, the core catcher device also reduced the length of the core to 0.95 m, and this was not taken into account when starting subsequent pushes. The resulting gaps are represented in the macroscopic charcoal record.

The upper 3.86 m of the Laguna Estero Blanco profile consists of dark (Munsell 10YR 3/1 to 2/1) organic-rich (20–80% on dry weight basis) sediments (Arford, in progress). The bottom 0.61 m of the core contains gray (Munsell 5Y 3/1) clay and gravels associated with the formation of the lake. For the macroscopic charcoal analysis, I sampled only the upper 3.86 m of sediment. The upper 0.05 m of sediment from each core section was regarded as unsuitable for analysis and therefore not included in my record. Because of the design of the core catcher, this material was likely accumulated from higher levels of the profile as the corer was moved into position for the next drive. These gaps are also represented in the macroscopic charcoal record.

Organic carbon concentration was relatively low (<40%) at the base of the core, but increased by ca. 7400 cal yr BP (>60%). Between 7400 and 3350 cal yr BP, the organic content remained high. From 3350 cal yr BP until present, organic carbon concentration was generally less than 30% (Arford, in progress).

Radiocarbon Analyses

Five AMS radiocarbon dates were obtained on charcoal or other small organic fragments. These assays yielded a normal stratigraphic sequence extending back to 7150 ± 40 ^{14}C yr BP (2 sigma range 7924–8031 cal. yr BP) (Table 3.1). Sedimentation rates were variable over the length of the core (Figure 3.5). Sedimentation was rapid (0.16–0.20 cm/yr) over the first ca. 2100 years of the record and slower (0.009–0.064 cm/yr) for most of the last ca. 5800 years of sediment accumulation. The sedimentation rate increased (0.39 cm/yr) during the most recent ca. 50 years of the record.

Charcoal Accumulation Data

A visual assessment of the charcoal concentration (particles/cm^3) and charcoal influx (particles/cm^2/yr) in the Laguna Estero Blanco sediment core reveals that charcoal concentrations are low and consistent for the lower 2.0 m of the core, ranging between 100–300 particles/cm^3 (Figure 3.6). The charcoal concentrations in the upper 1.85 m are higher and more variable, ranging between 100–1000 particles/cm^3. Charcoal concentrations in the upper 0.25 m are near zero, less than 50 particles/cm^3. Values for charcoal influx, which take into account the varying number of years represented by each

Table 3.1 Radiocarbon determinations on material from Laguna Estero Blanco sediment profile.

Lab Number [a]	Depth (m) [b]	Material Dated	$\Delta^{13}C$	Conventional ^{14}C Age (^{14}C yr BP)	Calibrated Age Range (cal. yr BP) $\pm 2\sigma$ [c]	Relative Area Under the Calibration Curve	Weighted Mean cal years BP [d]
AA-60651	0.38	Charcoal Fragments	-26.24	Modern	*	*	*
AA-60652	0.46	Charcoal Fragments	-26.02	682 ± 37	556–605 623–679	0.434 0.566	622
β-169602	0.63	Charcoal Fragments	-19.6	3140 ± 60	3170–3180 3210–3470	0.004 0.996	3353
β-176223	1.53	Charcoal Fragments	-25.4	5140 ± 40	5750–5829 5857–5947 5969–5987	0.375 0.575 0.050	5868
β-155065	1.92	Charcoal Fragments	-26.5	6080 ± 40	6761–6766 6795–6840 6849–7020 7081–7086 7129–7154	0.005 0.124 0.833 0.004 0.034	6927
β-155066	2.91	Wood Fragment	-29.7	6630 ± 50	7430–7578	1.000	7510
β-158437	3.78	Charcoal Fragments	-18.4	7150 ± 40	7865–7899 7924–8031 8093–8107	0.179 0.790 0.031	7959

[a] Analyses were performed by the University of Arizona AMS Laboratory (AA) and Beta Analytic Laboratory (β).
[b] Depth below sediment surface.
[c] Radiocarbon ages were calibrated using version 4.4.2 of the CALIB program (Stuiver and Reimer, 1993) and the dataset of Stuiver et al. (1998).
[d] The weighted mean of the calibration probability distribution was calculated from CALIB output.

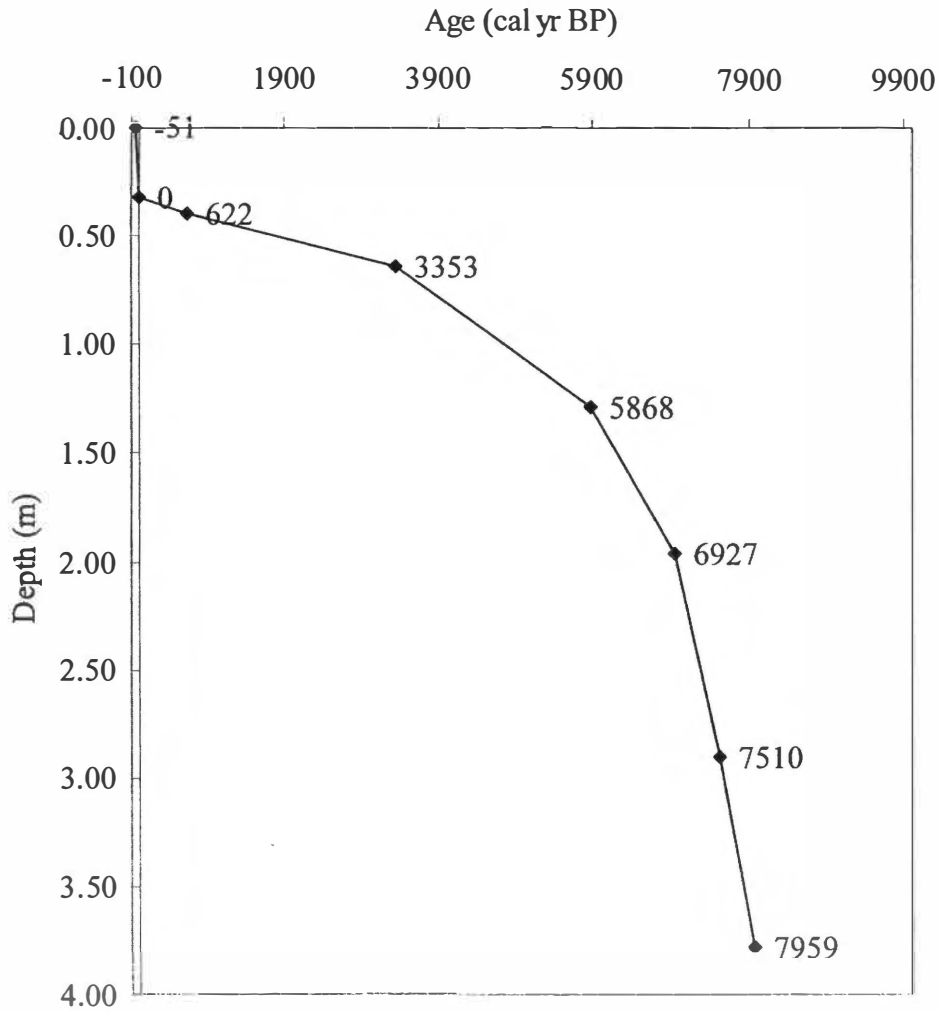


Figure 3.5 Age vs. depth relationship for Laguna Estero Blanco, Costa Rica. Model based on linear interpolation between the weighted means of the calibration probability distributions for the radiocarbon dates. The surface sediments of the core retrieved in 2001 date to -51 cal yr BP.

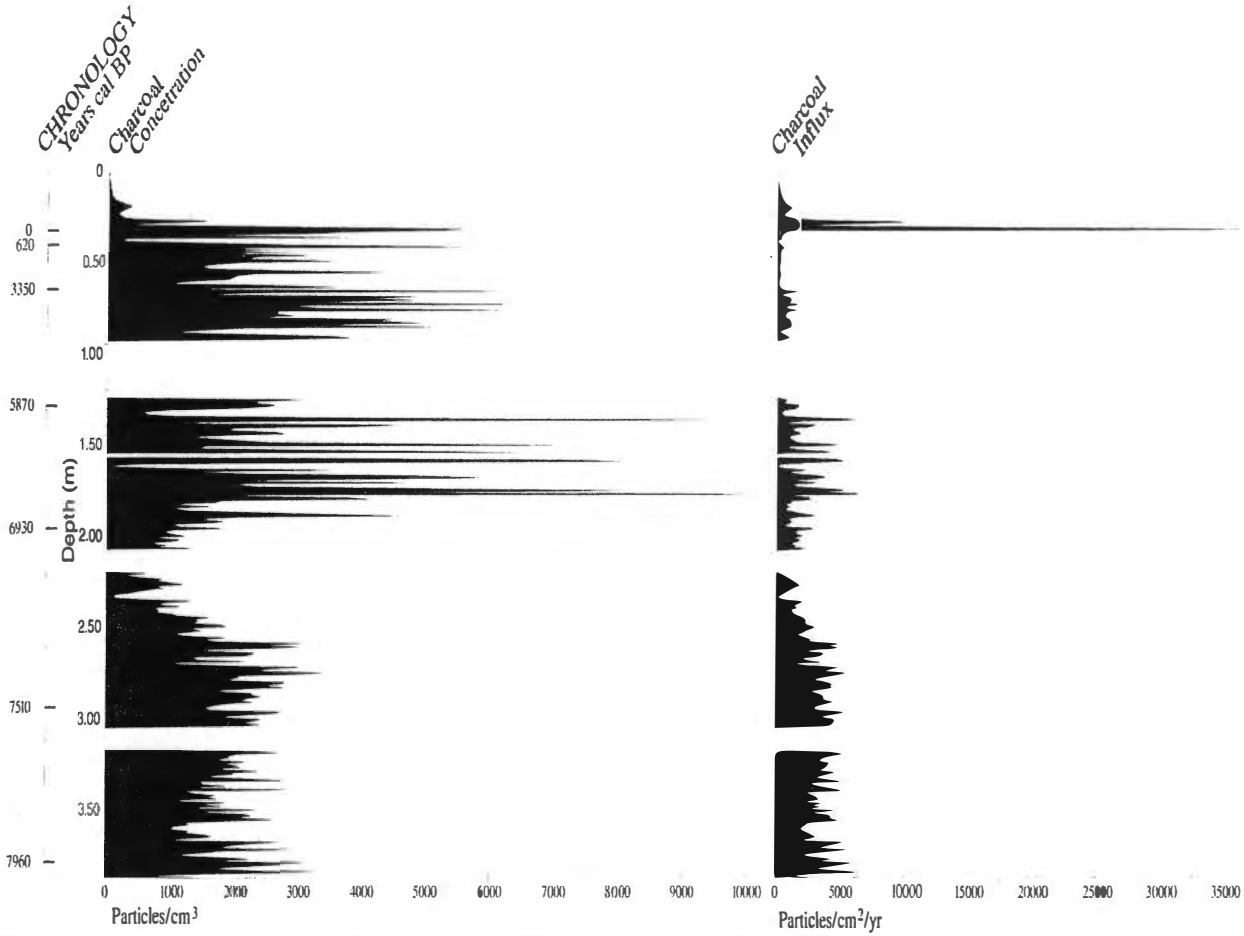


Figure 3.6 Macroscopic charcoal data from Laguna Estero Blanco, plotted by depth. Gaps occur where no core material was recovered. Figure shows stratigraphic column with radiocarbon dates, charcoal concentration (fragments/cm^3 wet sediment), and charcoal influx (fragments/cm^2/yr).

sediment sample, are higher and more stable in the lower 2.0 m of the core and generally lower but more variable for the upper 1.85 m of the sediment core.

Selection of Parameters for Analyzing Charcoal Influx Data

CHAR data from the Laguna Estero Blanco core were analyzed using a variety of background-window widths and peak-threshold ratios (Figure 3.7). The window width of 900 years over-generalized the data by obscuring changes in CHAR that are visually evident. The window width of 300 years matched the CHAR so closely that small fluctuations of the data within single peaks defined by each of the other window widths marked were identified as distinct fire episodes. The 450-yr and 600-yr window widths produced very similar results. Both resulted in almost identical numbers of peaks, with the only difference being the shorter peak time spans produced by the 450-yr window width. This narrowing of the peaks allows the fire episode to be more distinctly identified temporally. Therefore, a background-window width of 450 years was selected to analyze the Laguna Estero Blanco macroscopic charcoal record (Figure 3.8).

Three peak-threshold ratios were assessed to establish the best value for identifying peaks with a 450-year-background window width (Figure 3.8). These values resulted in 41, 29, and 20 peaks, respectively, for the length of the Laguna Estero Blanco macroscopic charcoal record. Previous studies using CHAPS were able to calibrate the peak-threshold ratio with dendrochronological data and modern fire regime estimates for northwestern North America (Long, 1996; Long et al., 1998; Millspaugh et al., 2000; Mohr et al., 2000; Long and Whitlock, 2002; Brunelle and Anderson, 2003; Brunelle and Whitlock, 2003; Long, 2003). No such information is available for the seasonally dry

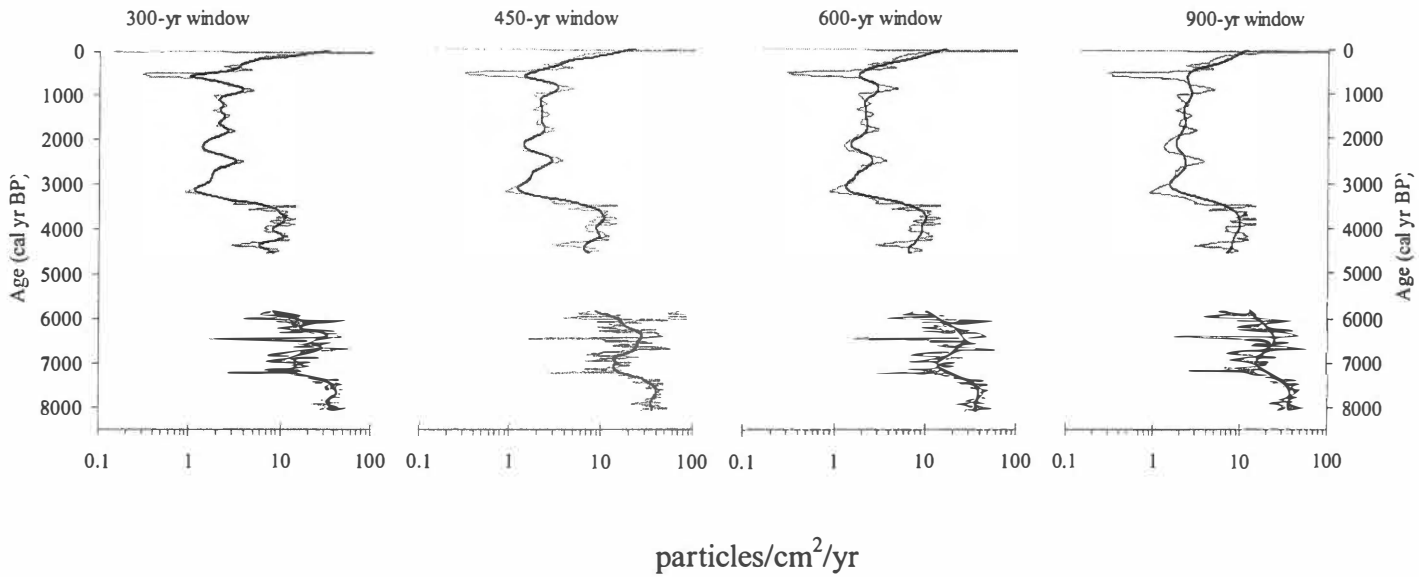


Figure 3.7 Comparison of background-window widths of 300, 450, 600, and 900 years to determine background levels of log-transformed CHAR for Laguna Estero Blanco. Black line represents the background level of influx defined by the window width.

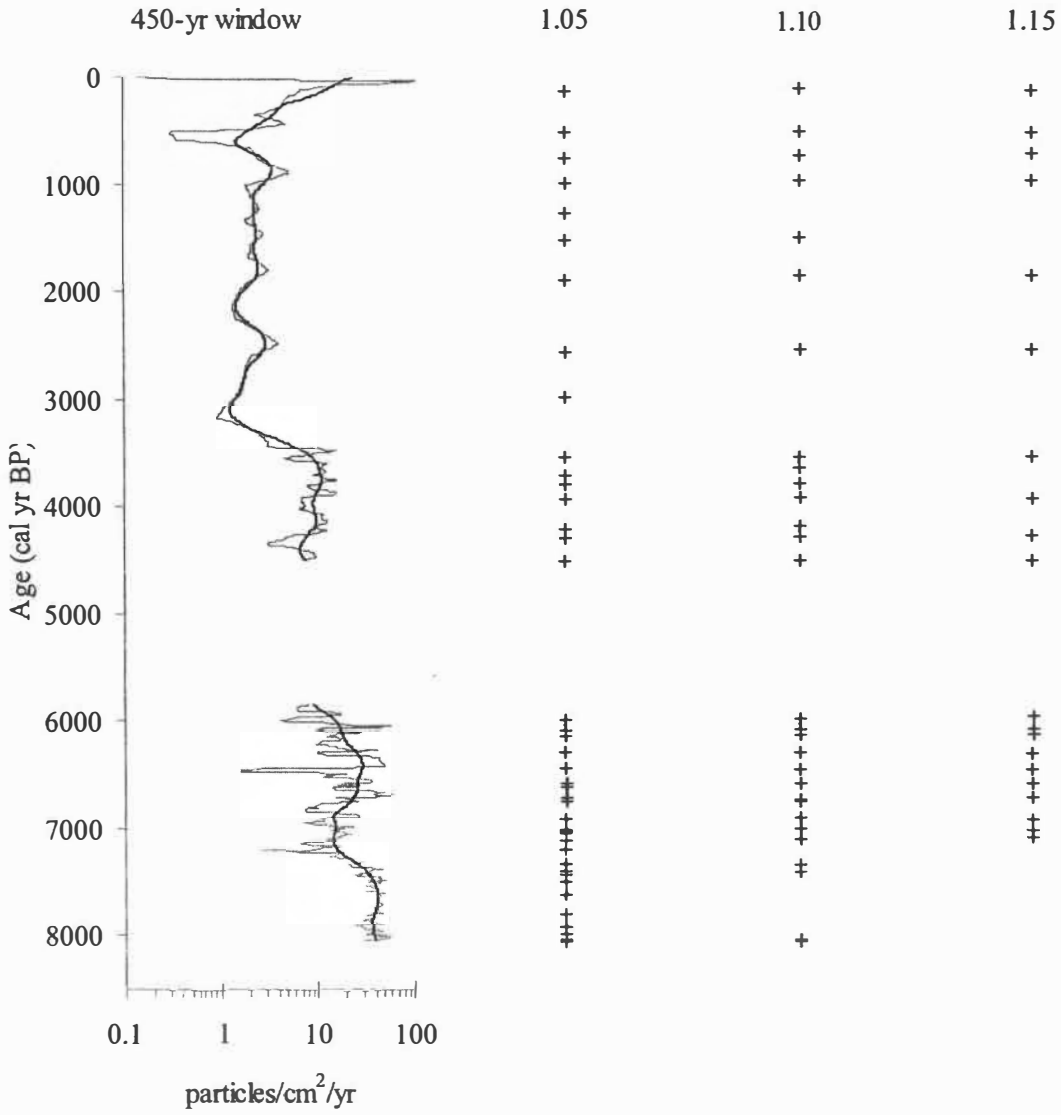


Figure 3.8 Comparison of peak-threshold ratios of 1.05, 1.10, and 1.15 using a background-window width of 450 years. Peaks (+) indicate a fire event.

tropical forests of Costa Rica. I had to select a threshold ratio based on previous studies from the very different northwestern temperate forest ecosystems, and on my best assumptions of what I thought was accurate.

The 1.05 threshold produced several peaks that consisted of only one level and that barely surpassed the threshold. This threshold ratio was rejected because the validity of these peaks seemed unlikely when compared to the influx values of other identified peaks. The 1.15 threshold failed to identify several peaks sustained for several levels that the other threshold ratios identified. Therefore, the ratio of 1.10 was selected as most favorable for identifying peaks of charcoal accumulation that represent fire episodes. This peak-threshold ratio is comparable to the threshold ratios most commonly used in northwestern North America (Long, 1996; Long et al., 1998; Brunelle and Anderson, 2003).

Macroscopic Charcoal Record

The CHAR data reveal the following fire history (Figure 3.9). Charcoal influx was high (30–40 particles/cm^2/yr) and fire events occurred with a low frequency (1–5 episodes / 1000 yr) from 8050–7400 cal yr BP. The increased fire frequency at the beginning of the record (Figure 3.9) may be an artifact of an end-effect inherent in the CHAPS. After 7400 cal yr BP the frequency increased and was sustained (5–10 episodes / 1000 yr) until approximately 3600 cal yr BP. Influx decreased slightly and ranged from 10–30 particles/cm^2/yr. During this period, the Mean Fire Return Interval (MFRI) ranged from 90–300 years. The MFRI is calculated by CHAPS as the number of peaks per 1000 years as calculated with a 1000 yr moving window. After about 3600 cal yr BP, influx

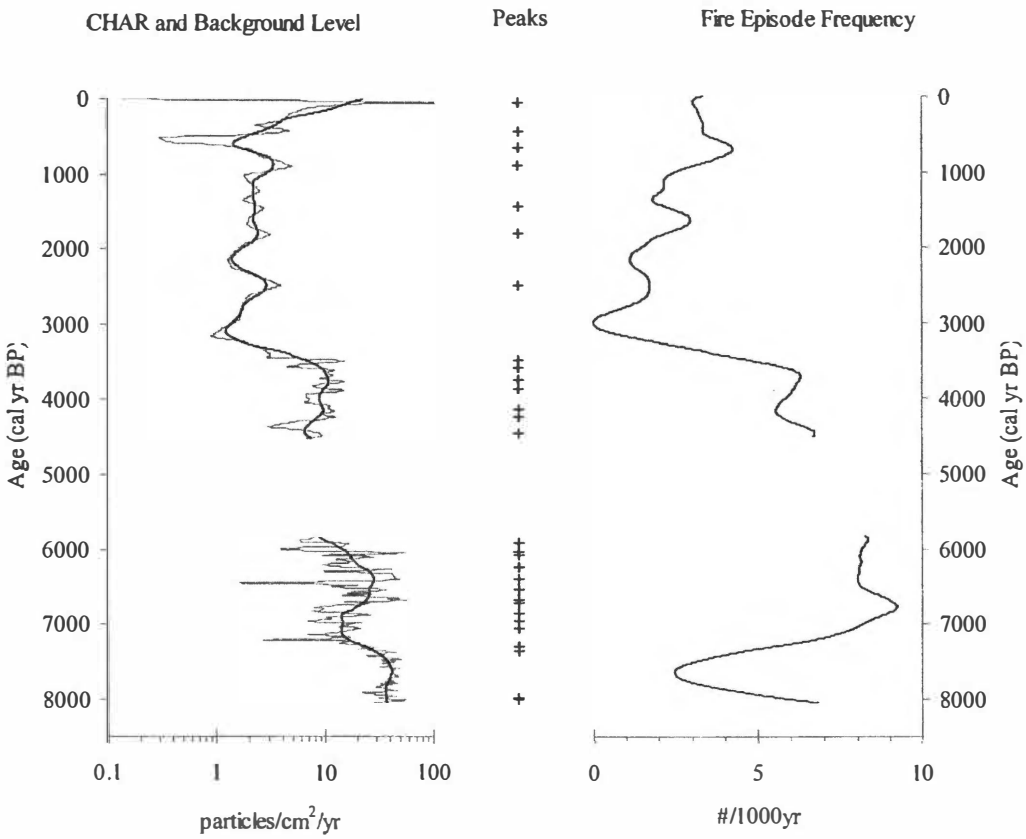


Figure 3.9 Log-transformed CHAR, peaks, and inferred fire frequency for Laguna Estero Blanco using a 450-yr window width and a 1.10 peak-threshold ratio. There are two peaks (+) almost on top of each other at the bottom of the peaks column.

(1–3 particles/cm^2/yr) and fire frequency (1–3 episodes/1000 yr) dramatically decreased. Influx increased to near 100 particles/cm^2/yr at about 100 cal yr BP.

Size Class Comparison

I compared the frequency peaks, within the different size classes into which I classified charcoal particles, to the total count (Figures 3.10 and 3.11). The same general trend is apparent in the three classes and the total count. The two larger size classes do show a rise in peak frequency at about 3600 cal yr BP. Also, due to the bulk of charcoal particles being in the smallest size class, the 250–500 μm size class bears the most resemblance to the total count.

Discussion

Chronology and Sedimentation

Sedimentation rates calculated from calibrated radiocarbon dates reveal changes in depositional environment during the period spanned by the Laguna Estero Blanco sediment profile. Following the formation of the lake basin by lahar and lava flows from an eruption 8000 yr ago, lower portions of the Laguna Estero Blanco core accumulated rapidly. This part of the core (3.86–4.47 m) consists of clays and gravels associated with the formation of the lake. Linear interpolation between the two lowest radiocarbon dates reveals that lacustrine sediments began to accumulate at a fast rate of 0.20 cm/year for the first ca. 500 years after lake formation. Sedimentation slowed slightly for the next ca. 500 years and abruptly decreased at about 6900 cal yr BP. It continued to decrease until an abrupt increase in sediment accumulation in modern times.

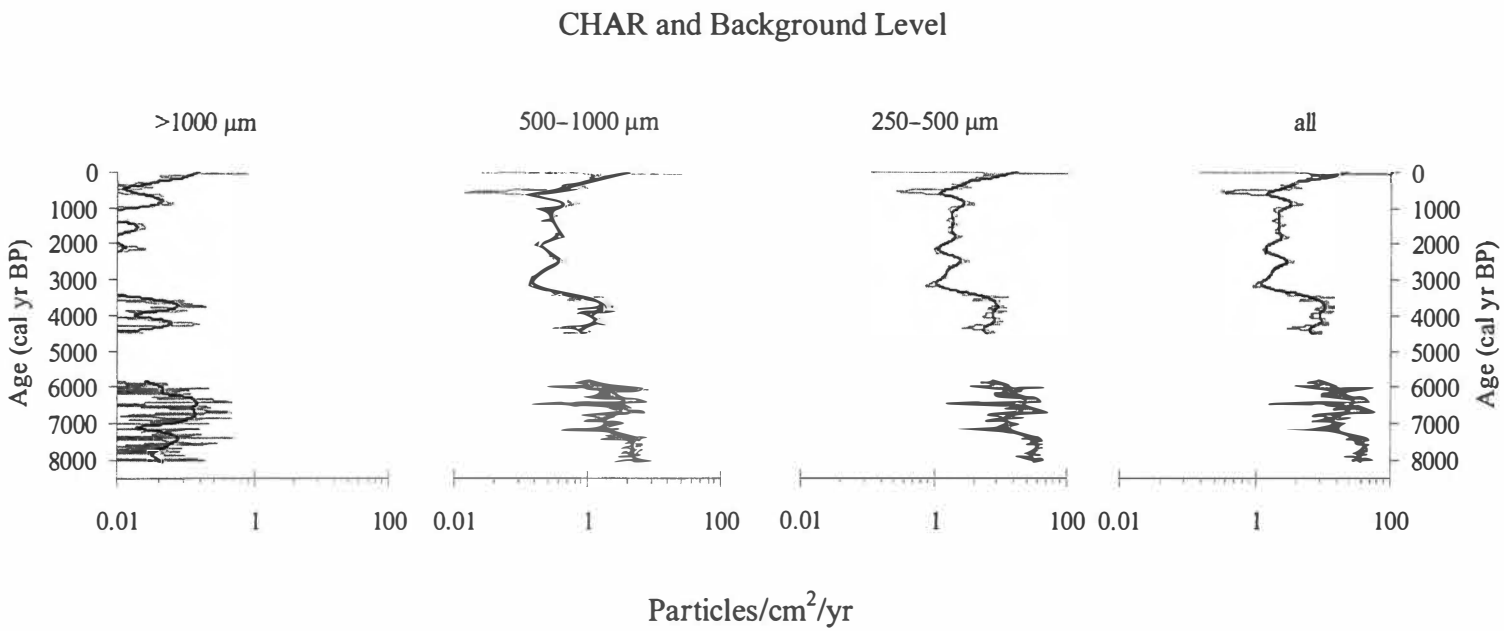


Figure 3.10 Comparison of charcoal influx based on different size classes of macroscopic charcoal.

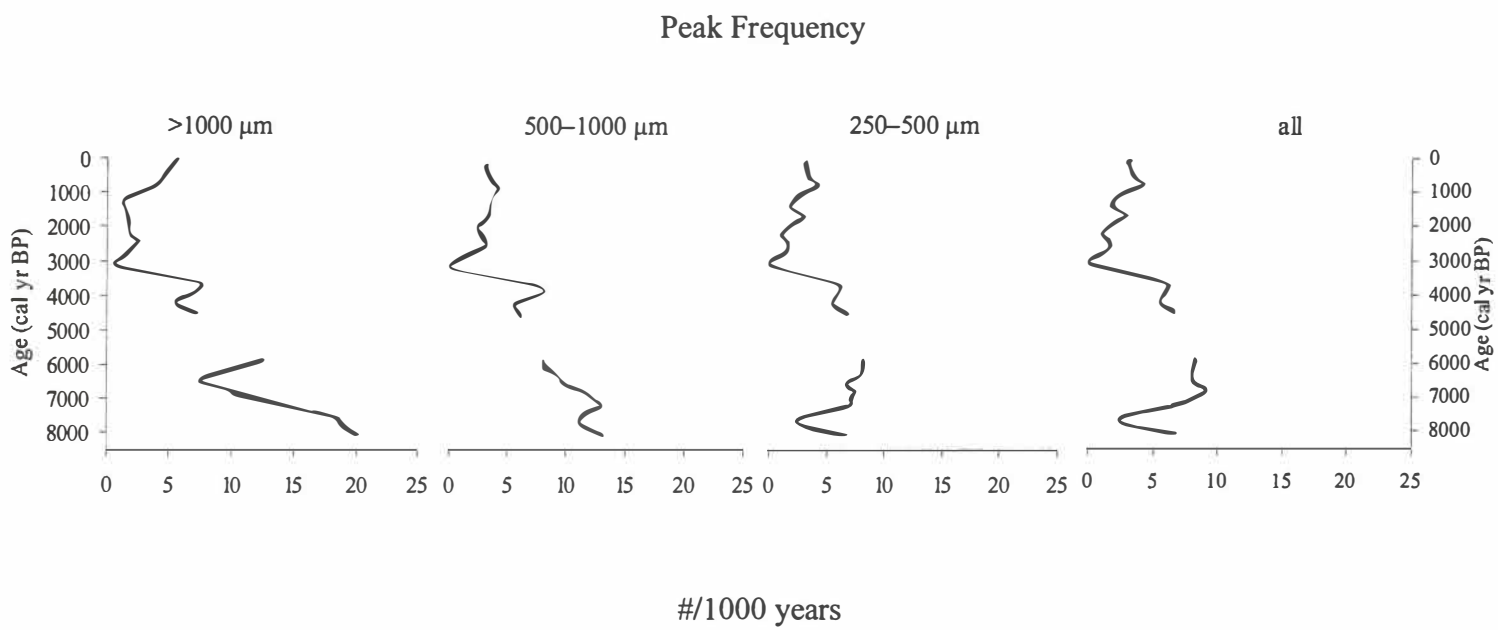


Figure 3.11 Comparison of inferred fire frequency based on different size classes of macroscopic charcoal.

The rapid initial sedimentation is likely associated with erosion of a barren landscape after volcanic activity approximately 8000 cal yr BP. Erosion would be expected to decrease as plants colonize the lava and lahar flows. The change in sediment accumulation at about 6900 cal yr BP may indicate stabilization of the vegetation surrounding the lake. I take the dramatic increase in sedimentation during modern times to be associated with the conversion of the watershed into its present day condition of cattle pasture. As a result of these changes in sedimentation rates, the lower half of the sediment core represents approximately 1000 years, while the upper half of the sediment core represents approximately 7000 years. The upper meter of sediment alone represents on the order of 5000 years. These very large changes in sedimentation rates over the period spanned by the core made converting the charcoal data into pseudo-annual values very helpful for interpreting the data.

The Laguna Estero Blanco pollen and microscopic charcoal records reveal the following paleoenvironmental history (Arford, in progress). The first ca. 1000 years of the record is dominated by trees. Microscopic charcoal concentrations during this period are uniformly low. Grass pollen and trilete fern spores, most likely indicative of the disturbed conditions following the formation of the lake, show a high percentage (upwards of 80% of total pollen) of trees until about 6900 cal yr BP. Arford attributes a subsequent decrease in tree pollen, a sharp rise in microscopic charcoal, a rise in grass pollen, and a change in sedimentation rate to a possible mid-Holocene drying event. He suggests that the change in sedimentation rate may indicate the possibility of missing sediment due to the desiccation of the lake and sediment being removed by aeolian processes. He suggests that an increase of *Myrica* pollen immediately prior to this abrupt

change indicates mire formation associated with a drying of the lake system. By approximately 5800 cal yr BP, the forest has recovered from the disturbance event. Arford proposes that a peak in microscopic charcoal, increase in grass pollen, an abrupt decline in tree pollen, and the presence of *Zea mays* subsp. *mays* pollen starting at ca. 3300 cal yr BP signals anthropogenic disturbances within the Laguna Estero Blanco watershed associated with prehistoric agriculture. Arford argues that this human signal dominates the remainder of the record as grass pollen continues to be dominant, tree pollen has its lowest percentages of the entire record, and *Zea mays* subsp. *mays* pollen is found in a majority of all samples younger than 3300 cal yr BP.

Interpretation of Charcoal Record

This high-resolution macroscopic charcoal record matches well with the low-resolution microscopic charcoal and pollen records (Arford, in progress). Curves of macroscopic and microscopic charcoal accumulation are almost identical. The two fire episodes at the base of the record could reflect fires associated with the volcanic activity that formed the lake (Figure 3.9). The peaks in charcoal that define the fire events are supported by correspondingly high percentages of grass pollen and trilete fern spores. Following these peaks, fire episodes are few for the first 1000 years of the record. This period corresponds with the stable, tree-dominated vegetation indicated by the pollen record.

Fire frequency reached a maximum at about 6800 cal yr BP. This increase corresponds to a peak in microscopic charcoal, a rise in grass pollen, and a decrease in tree pollen that Arford (in progress) hypothesizes indicates a dry period at Laguna Estero

Blanco. After this peak, fire frequency declines until about 3600 cal yr BP. This peak in fire frequency occurs slightly prior to the first appearance of *Zea mays* subsp. *mays* and may provide a signal of earlier human presence at Laguna Estero Blanco. This signal is more evident when the data are examined by charcoal particle size class. This increase in fire frequency is more apparent in charcoal particles greater than 500 μm in maximum dimension. Because larger charcoal particles (>500 μm) have been shown to be transported only a very short distance (Ohlson and Tryterud, 2000), I suggest that the peaks in charcoal within the >1000 and 500–1000 μm size classes at this time indicate burning on the very shores of Laguna Estero Blanco, most likely associated with human agriculture. This interpretation is consistent with archaeological evidence of human occupation in the area as early as 3500 ^{14}C yr BP (Norr, 1982–1983), and with the presence of maize and other indicators of agricultural activity at the other Miravalles lakes at this time (Arford and Horn, 2004; Arford, in progress).

Macroscopic charcoal concentration and influx decrease after 3600 cal yr BP and remain low until about 400 cal yr BP. I suggest that this lull in fire activity represents an agricultural period occurring after the initial conversion of the forested areas surrounding the lake into agricultural fields. *Zea mays* subsp. *mays* pollen grains identified by Arford indicate that humans were indeed growing crops on the shores of Laguna Estero Blanco. The low charcoal values indicate that new lands were not cleared using fire after the initial forest conversion and that pre-Columbian agriculture near the lake did not involve repeated burning to maintain fields. Only sparse use of fire in agriculture could explain lower charcoal concentration and influx rates.

I suggest that the increase in charcoal influx approximately 200 cal yr BP is associated with disturbance related to European settlement. In recent times, development of cattle pastures has been the principal cause of deforestation of forests throughout Mexico and Central America (Stern et al., 2002). Initial creation of pastures often involves the use of fire. Fire also is used as pasture management to maintain forage composition, productivity, and quality in neotropical dry forest environments (Stern et al., 2002; Kauffman et al., 2003). Along with the introduction of cattle to the ecosystem, the invasive African grass jaragua (*Hyparrhenia rufa*) has spread throughout pasturelands in the neotropics (Allen, 2001; Stern et al., 2002) adding to the available fuel for fires (Allen, 2001; Janzen, 2002).

Comparison with Regional Studies

The macroscopic charcoal record from Laguna Estero Blanco shows some similarities to the microscopic charcoal record from Lago Cote (Arford, 2001). The Lago Cote record reveals abundant charcoal particles in the basal zone from 4000 cal yr BP to 2600 cal yr BP. Arford (2001) attributed these high charcoal abundances to anthropogenic fires. The sharp drop in charcoal abundance at 2600 cal yr BP was attributed to a change to a wetter climate and a possible population decline. This decline in charcoal occurs 1000 years later at Lago Cote than at Laguna Estero Blanco, but in both cases the pattern is one of higher charcoal values during the early part of the late Holocene followed by lower charcoal values during the later part of the late-Holocene period. It is important to note, however, that a macroscopic charcoal record and a microscopic charcoal record may provide different signals (local vs. regional) and may

reveal differences in the timing of those signals. An increase in regional burning does not necessarily indicate an increase in local burning, and vice versa.

On the other hand, the macroscopic charcoal record from Laguna Estero Blanco shows patterns opposite of those in a macroscopic charcoal record from Lago de las Morrenas 1, on Cerro Chirripó in the Cordillera Talamanca, Costa Rica (League and Horn, 2000) (Figure 3.1). League and Horn (2000) found greater influx of charcoal during the later Holocene (from 4200 ^{14}C yr BP – present) and lower charcoal influx between 6800–4200 ^{14}C yr BP. They concluded that the low influx values were a result of wetter conditions that made fires infrequent in the páramo and that increased influx following 4200 ^{14}C yr BP may be linked both to drier climate and to increased human population density in the nearby foothills. The stark contrast between these two charcoal records may be a function of differing climate histories and/or fire-climate relationships. Perhaps the general climate change at these two sites was out of phase. Another possibility is that the same general climate history prevailed but that the effect of climate on the fire regimes differs greatly at these two sites. The treeless vegetation of the high-elevation páramo differs markedly from the tree-dominated forests of the seasonally dry tropical lowland forests of northwestern Costa Rica. Changes in the total precipitation and/or the seasonality of rainfall should likewise influence fire history in these ecosystems differently.

Many sediment records of terrestrial plant fossils (pollen, plant macrofossils, and microscopic and macroscopic charcoal) from the circum-Caribbean region are strongly influenced by human activity. One record that has been put forward as an indicator of Holocene climate is from a marine sediment core from the Cariaco Basin (Haug et al.,

2001). Haug et al. (2001) argued that titanium variations provide a direct measure of rainfall and runoff from the Orinoco River in northern South America. High titanium abundances indicate increased terrigenous input to the Cariaco Basin and imply increased rainfall and increased runoff from the watersheds of rivers that drain into the basin (Peterson et al., 2000). The titanium record indicates wetter conditions from 8000–4000 cal yr BP followed by drier conditions during the later Holocene. As an explanation, the authors posited that changes in the seasonality of insolation have affected the mean latitudinal position of the Atlantic Intertropical Convergence Zone (ITCZ) over the course of the Holocene.

The resemblance in the general trends of the macroscopic charcoal record from Laguna Estero and the bulk titanium curve from the Cariaco Basin (Haug et al., 2001) is suggestive of a common forcing mechanism (Figure 3.12). The migration of the ITCZ is a dominant control of Holocene variations of precipitation in the region. The more northerly migration of the ITCZ during the middle Holocene, caused by greater seasonality of northern hemisphere insolation (Figure 3.13), may have increased wet-season rainfall at Laguna Estero Blanco. This climate interpretation is in line with the interpretation of League and Horn (2000) even though their fire history is opposite my record. Many other records from the wider circum-Caribbean also suggest wetter climate in the middle Holocene (Curtis et al., 1998; Curtis et al., 1999; Higuera-Gundy et al., 1999; Brenner et al., 2001; Fritz et al., 2001).

My record reveals that the time interval of 8000–4000 cal yr BP, which was presumed to be wet in other areas of the circum Caribbean, was associated with greater

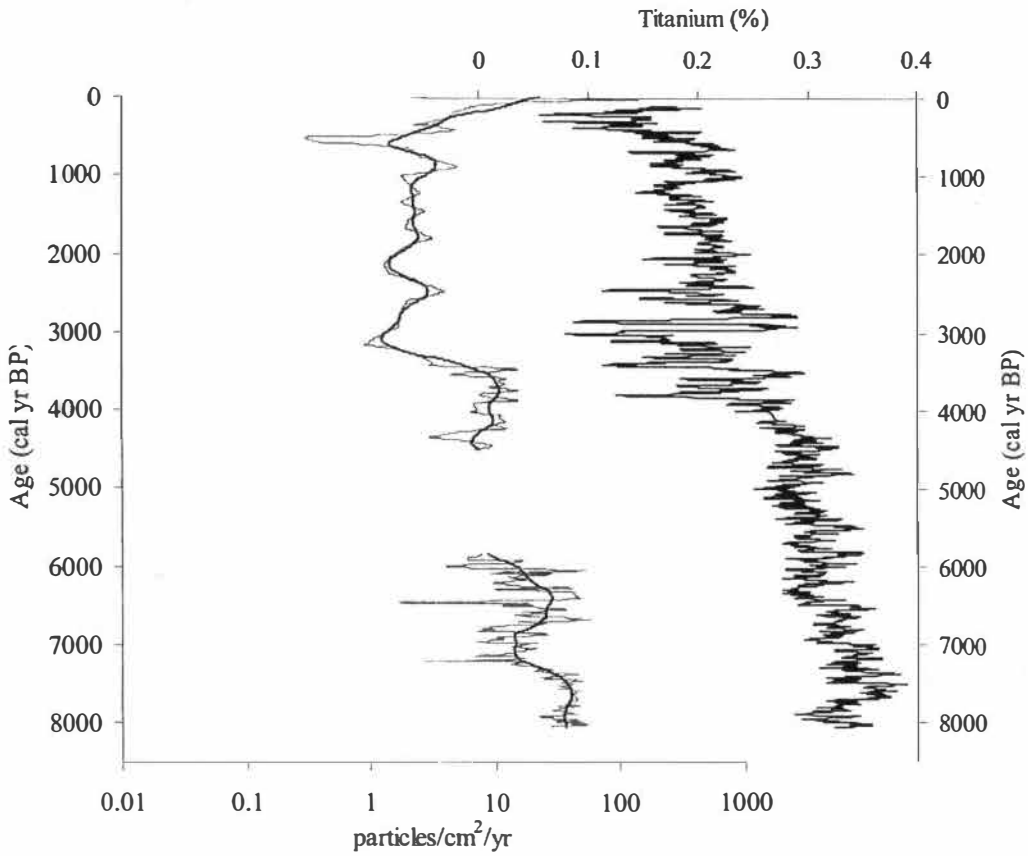


Figure 3.12 Comparison of Laguna Estero Blanco macroscopic charcoal influx with % bulk titanium (Haug et al., 2001). Titanium is a proxy for rainfall; greater % corresponds to wetter conditions.

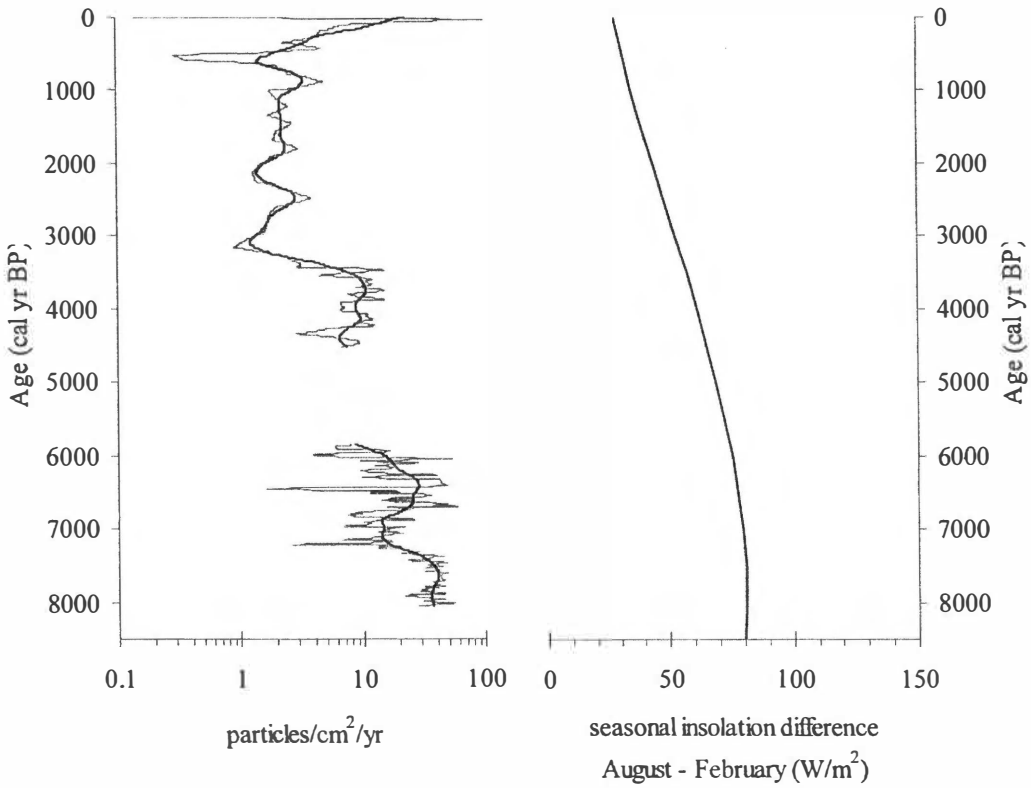


Figure 3.13 Comparison of Laguna Estero Blanco macroscopic charcoal influx with seasonal insolation difference (August minus February) at 10 °N. Original data from Berger (1978).

fire activity at Laguna Estero Blanco and that the inferred drier conditions of 4000–0 cal yr BP are associated with decreased fire activity at Laguna Estero Blanco. While this may seem counter-intuitive, this situation has been reported previously (Higuera-Gundy et al., 1999) and may be explained by changes in seasonality. Increased seasonality in insolation during the middle Holocene (Figure 3.13) may have resulted in wetter wet seasons and drier dry seasons at Laguna Estero Blanco. If so, the increased precipitation during the wet seasons would have allowed more vegetation to grow, providing more fuel for burning during the dry seasons (Curtis et al., 1999; Higuera-Gundy et al., 1999). During the late Holocene, decreased seasonality may have led to drier wet seasons and milder dry seasons, possibly decreasing both fuels and opportunities for fires.

Conclusions

I suggest that fire during the past 8000 years at Laguna Estero Blanco has ultimately been controlled by precipitation and seasonality. However, two periods deviate from this pattern related to human disturbances. The first occurs at about 3600 cal yr BP. The initial deforestation by pre-Columbian peoples caused an increase in macroscopic charcoal in the sediment record. Prior to this period, fire episode frequency was decreasing in parallel with the titanium record. The increase of fire episodes at this time is a human-caused departure from the climatic control that was previously evident. The second deviation is during modern times. European introduction of cattle and the introduction of jaragua to the landscape have altered the fire regime from what it naturally and climatically would be.

The idea of restoring/recreating seasonally dry tropical forests by removing fire is an accepted environmental strategy in northwestern Costa Rica (Allen, 2001; Otterstrom, 2004). This approach was developed without long-term paleoecological records. The macroscopic charcoal record from Laguna Estero Blanco, together with the pollen and microscopic charcoal record of Arford (in progress), reveals that fires were more frequent during the heavily forested, and possibly wetter, middle Holocene.

The establishment of jaragua has changed the fire regime of seasonally dry tropical forests in northwestern Costa Rica. This change appears to be evident in my macroscopic charcoal record. The removal of the jaragua is a key issue for restoring the native forests. While the suppression of fire to achieve this goal may be an appropriate short-term strategy, policymakers should think about conservation and restoration of the ecosystem with an eye toward its long-term history.

In addition to the recent introduction of jaragua, vegetation has changed dramatically in the last 8000 years. Policymakers must also ask themselves what type of vegetation assemblage are they trying to restore and whether such a goal realistically be accomplished. Are they trying to restore the less frequently burned, more open mix of forests and grasslands that characterize the late Holocene, or the more frequently burned, more heavily forested ecosystem of the early Holocene? Can either goal actually be achieved with the climatic and anthropogenic influences that currently exist in northwestern Costa Rica?

Increasing the numbers of paleoecological studies in these forests will provide more evidence and a greater spatial resolution of data for developing better conservation strategies. Policymakers must also understand that because climate seems to be the main

fire control, as climate changes so will the fire regime. Long-term paleoecological studies, such as this one, can provide both researchers and policymakers with evidence on how such changes have taken place in the past. While not a frequent phenomenon, fire has been a part of the forests of northwestern Costa Rica for the past 8000 years. Because the past is a window into the future, fire will continue to be a part of this ecosystem.

CONCLUSIONS

As the first step in my research, I investigated different methods for isolating and quantifying macroscopic charcoal particles in Holocene lake sediments from Costa Rica. I sought a method that quickly disaggregated the sediment samples without damaging charcoal fragments because I wanted to improve visual identification and quantification of charcoal particles by making it easier to distinguish charcoal particles from dark, non-charred organic matter. After reviewing the literature and conducting a series of methodological experiments, I concluded that a method of treating samples with 3% U.S.P. cosmetic grade H_2O_2 for a period of 24 hours provided the best preparation for macroscopic charcoal analysis for my study.

This part of my research also revealed some issues regarding macroscopic charcoal analysis techniques. Two series of charcoal counts based on contiguous sediment samples subjected to identical treatments showed similar general trends but peaks did not always match and absolute values of particles were variable. This is a finding that warrants more attention. It may be necessary to use larger sample volumes and count more charcoal fragments per level to achieve true replicability. This is especially important when using statistical analyses (i.e., CHAPS) that seek to precisely identify individual fire peaks. Perhaps using larger sample volumes could reduce the small-magnitude variability that exists between duplicate analyses. More research on macroscopic charcoal analysis techniques is needed to better understand this technical issue, especially when so many different techniques exist.

The second part of my research was part of a larger project that includes paleoecological analyses of sediment cores from six lakes on the lower southwestern slope of Volcán Miravalles, in the Cordillera de Guanacaste in northwestern Costa Rica. I sampled and sieved a sediment core from Laguna Estero Blanco at contiguous 1-cm intervals to produce a high-resolution charcoal record. I found that fire has been a part of the ecosystem for 8000 years. Charcoal concentration and influx were greatest for the period approximately 8000–3600 cal yr BP. An increase in inferred fire frequency at about 3600 cal yr BP coincides with archaeological evidence (Norr, 1982–1983) and palynological evidence (Arford, in progress) of the earliest human occupation of the slopes of Volcán Miravalles, and with drier climatic conditions. After this time, charcoal concentration and influx and fire frequency decreased until modern times. I concluded that an increase in sedimentary charcoal in the last several centuries corresponded to recent conversion of the landscape from forest to cattle pasture by European settlers.

Using CHAPS with the Laguna Estero Blanco macroscopic charcoal records proved helpful in my analyses. The programs allowed me to statistically dissect the data by time rather than depth, as has been done in previous macroscopic charcoal studies in Costa Rica (League and Horn, 2000; Anchukaitis and Horn, 2005). This was very helpful because of the extremely slow sediment accumulation in the upper half of the Laguna Estero Blanco sediment core. Converting the data into pseudo-annual values allowed me to interpret changes in the charcoal that I might have not seen otherwise. However, this conversion also introduces an unknown amount of error into my record. Extrapolated changes in charcoal deposited during approximately 5000 years in the upper

meter may not be precise because actual sedimentation rates were probably not uniform between dated intervals, as I assumed in interpolating dates.

Another potential limitation of using CHAPS was not having dendrochronological data available for calibrating my peak-threshold ratio to known, precisely-dated fire events. Studies using CHAPS in the Pacific Northwest have had this calibration technique available, allowing the researchers to have confidence in their selected peak-threshold ratios (Long, 1996; Long et al., 1998; Brunelle-Daines, 2002; Long and Whitlock, 2002; Briles, 2003; Long, 2003). I believe that my selected peak-threshold ratio was acceptable for my record, but having a dendropyrochronological record could have reinforced my threshold selection.

Overall, my research produced a new method for preparing macroscopic charcoal particles that was better suited for my study than any techniques that previously existed. This method has yielded samples that should be well suited for image analysis of charcoal particles. Each of my samples has been archived in the Laboratory of Paleoenvironmental Research at the University of Tennessee for future analysis.

My research also has shown that fire has indeed been a part of the seasonal tropical lowland forest ecosystem for the past 8000 years. While anthropogenic fires cannot be distinguished from natural fires in my record, it is unlikely that humans have been causing fires near Laguna Estero Blanco for all of the last 8000 years. Volcanism and lightning have probably ignited fires as well as human activity. This evidence contradicts previous assessments of the role of fire in seasonally dry tropical lowland forests as unnatural (Murphy and Lugo, 1986; Janzen, 1988; Tenenbaum, 1994; Janzen, 2002). An increase in the frequency of fire events coincident with palynological

evidence of initial human occupation of the site (Arford, in progress) suggests that humans may have altered the existing natural fire regime. The increase in charcoal influx in modern sediments indicates that modern land uses have further altered the fire regime. Unfortunately, changes in human population densities or pre-Columbian land uses cannot be inferred from changes in the macroscopic charcoal record. However, the record does reveal information about pre-Columbian fire use that may be of archaeological significance. Specifically, it reveals that pre-Columbian agriculture may not have involved extensive use of fire following initial forest clearance.

Changes in the macroscopic charcoal record do appear to be parallel with changes in the pollen record, but I cannot determine whether changes in the fire regime affected the vegetation or whether changing vegetation caused changes in the fire regime. The macroscopic charcoal record bears some resemblance to an inferred regional precipitation record (Haug et al., 2001) showing trends that have been observed elsewhere in the neotropics (Curtis et al., 1999; Higuera-Gundy et al., 1999). Macroscopic charcoal influx was greater in the early to middle Holocene (8000–3600 cal yr BP) when precipitation and seasonality were greater. Charcoal influx at Laguna Estero Blanco decreased during the late Holocene when precipitation and seasonality were lower. These same climate trends, but with an opposite effect on fire, were seen by League and Horn (2000) at their site in the Cordillera de Talamanca of southern Costa Rica.

This research has added a valuable long-term fire history record to northwestern Costa Rica. This record reveals how fire has been a part of an ecosystem for at least 8000 years when it was previously thought to be non-existent. Long-term records such as this should enable scientists and policymakers to gain a better understanding of the

ecology of seasonally dry tropical lowland forests in Central America and help them find ways to best preserve this important ecosystem. While the suppression of fire to remove jaragua from this ecosystem may be an appropriate short-term approach, policymakers must develop conservation and restoration policies that take into account long-term interactions among fire, climate, and humans. With a better understanding of these interactions, we will have a better idea of what we are managing for, especially in the context of future environmental changes.

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VITA

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