Characterization and chronology of charcoal found in the volcanic ashfall that impacted a late Valdivia community in coastal Ecuador

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ABSTRACT. Several samples of fossilized wood (charcoal) were collected in the Papayita archaeological site, in coastal Ecuador. This carbonized material was encountered inside a layer of volcanic ash that sealed the site. The ash-sized tephra was produced by a sub-Plinian eruption from the Guagua Pichincha volcano contemporaneous with the late Valdivia phases during the Formative Period. Each of the samples was sectioned into 10 to 15 subsamples and examined under a Scanning Electron Microscope (SEM), producing high-resolution images with a large depth of field where the anatomical structures and their geochemical composition were vividly discernible. Each sample corresponds to organic matter of vegetable origin, that is, carbonized wood in the form of small rocks, whose appearance is that of carbonized woody tree trunks and or branches. We were able to observe vascular structures, specifically bundles of xylem. It was possible to conclude that these tracheids underwent a physicochemical transformation typical of petrification processes, leaving the molds intact. This allowed us to determine structural elements that support the identification of the group of plants to which these samples belong, through the methodology of comparison of the anatomical components of current species. The fossilized wood structures are three-dimensional and present characteristics that correspond to the group of higher plants, Gymnosperms, of the Podocarpaceae group. Among them, quadrangular tracheids, circular hole-shaped pits in the vascular system, and absent resin canals stand out. Central to the analysis is the presence of transverse parenchyma, which can be ascertained to correspond to vegetation from climates that are temperate or cold.

KEYWORDS: Charcoal, petrification, late Valdivia, Formative period, Paleoxylology, volcanic ash, Guagua Pichincha Volcano

INTRODUCTION

Fossilization or petrification is a process that transforms the meristematic, parenchymatic or soft and sclerenchymatic or timber tissues of plants composed specifically of carbon, oxygen and hydrogen (BoSSHARD, 1955; HANSEN and WRIGHT, 1999; GIRI et al., 2004; SHAH et al., 2017). These elements convert cellulose, hemicellulose, lignin and suberin into solid material or rock, composed predominantly of silica, calcium carbonate and other minerals (Rumpel et al., 2002; HIBBETT et al., 2016; GIANNOTAS et al., 2021). Charcoal is similar to
latter materials representing foliated wood as a result of volcanic eruptions (Asouti, 2003; Titiz and Sanford Jr., 2007; Miyabuchi et al., 2012; Moser et al., 2018). As magma fragments into volcanic ash, it can cause the combustion of wood to some degree, and facilitate the transport of this wood, now as charcoal, from trees that were originally situated atop or near the volcano (Belousov et al., 2007; Scott, 2010; Glasspool and Scott, 2013). This preserved material due to charcoalification may reveal fundamental indications about the origin of the native flora coming from the wood and the timing of volcanic eruptions (Hatcher, 2002; Scott and Glasspool, 2005; Scott et al., 2008). Consequently, characterizing the contemporary flora surrounding the volcano during its eruptive phase is feasible, and concurrently, the same material can provide evidence of the timing of the volcanic eruption through the utilization of the C14 dating method (Vogel et al., 1990; Orsi et al., 1996; Harangi et al., 2010; Wild et al., 2010).

A fundamental factor to consider is the size of the fragments or samples found, since these are directly related to the distance from the origin to the place where they were found (Swain, 1973; Villa, 1982; Clark, 1988). In other words, if the traveling was long, the fragments, as in this study, are small, and on the contrary, if there is almost no or little transport, the fragments or pieces of petrified wood will be larger. Therefore, it can be predicted whether such plant remains are, due to their characteristics, plants of the given region or not (Fedoroff et al., 1990; Zhang et al., 2011; Im et al., 2012). Studies on petrified plants, fossil or carbonized wood are of interest in different study fields such as geology, paleobotany, ecology and climate change (Poole, 2000; DiMichele and Gastaldo, 2008; Taylor et al., 2009). Petrified wood and charcoal are observed worldwide in various geographical areas in which the presence of pyroclastic sediments or volcanic ash, through hydrolysis, produce silica minerals in solution and other elements or hydrothermal sources main component of which is dissolved silica (Chester et al., 1987; Hatipoğlu and Türk, 2009; Cardenas et al., 2014; Zhang et al., 2014).

New excavations of the Papayita site in the Province of Manabí, coastal Ecuador, have revealed significant archaeological evidence indicating domestic activities, small garbage middens, stone tool manufacture, a hearth, and human skeletal remains, among other things, all associated to the late Valdivia cultural tradition of the Ecuadorian Formative Period. The cultural remains of this single component site, are covered with a layer of volcanic ash which included fossilized wood in the form of charcoal. We have taken samples from these volcanic deposits in order to characterize the origin of the wood and its importance in relation to both, the volcanic eruption and the human and cultural context of its time. Therefore, it can be predicted that these plant remains can be characteristic of the plants in their volcanic zones of origin. The data from this study may have a very significant scope, since the ash buried this Valdivia settlement, and left an unprecedented devastation in coastal Ecuador some 3,500 years ago.

**STUDY AREA AND HISTORIC CONTEXT**

The Valdivia Culture has been named after the site of its discovery at Valdivia on the coast of Ecuador. This culture is considered to be one of the oldest and most complex ceramic cultures of the Americas. It is a significant area of archaeological research due to its early development and influence on subsequent cultures in the region. It dates back to the Early Formative period of pre-Hispanic Ecuador where it existed between 3800 and 1450 BC (Zeidler and Ubelaker, 2021). It is characterized by one of the earliest ceramic traditions in the Americas, by the beginning of sedentary village life, and the cultivation of domesticated plants. Valdivia sites can be found across much of the western coastal lowlands of Ecuador (Lathrap et al., 1977; Rowe and Duke, 2020). The Valdivia culture was first identified and dated by Meggers et al. (1956) in the late 1950s/early 1960s based on the pottery typology and radiocarbon dates from excavations at sites such as Valdivia, Buena Vista, Palmar, and Punta Arenas. Initially, researchers posited claims of a transpacific Japanese Jomon source for the Valdivia pottery of Ecuador (Estrada and Meggers, 1956). This hypothesis has since been strongly dismissed by evidence for an in situ Valdivia evolution (Lathrap et al., 1975; Lathrap et al., 1977).

Besides the longevity evidenced by the duration of the Valdivia tradition of almost
2400 years, several aspects make its study both fascinating and controversial. As mentioned previously, its origin continues to generate research and productive debates (e.g. Kanomata et al., 2016; Zubova and Ras, 2018). Its ending, however, is also of interest to researchers as stated from very early on by the pioneering work of Hill when she wrote: “How Valdivia ended is no clearer than how it began” (Hill, 1972). Zeidler and Pearsall’s groundbreaking studies in the Jama valley have provided some provocative hypotheses, among them the possibility of major social and cultural changes happening in the later part of the Valdivia tradition due to major environmental transformations. Among the probable ones are Andean volcanic eruptions that affected the coastal regions of Ecuador during the late Formative (Isaacson, 1994; Zeidler, 1994). It is precisely within this time period and with evidence of a major volcanic ashfall sealing the late Valdivia component, that we have documented the archaeological information coming out of the Papayita site (Fig. 1).

Papayita is a single-component, such as one house settlement situated east of the town of Picoazá and west of the bustling city of Portoviejo, in the south-central region of the Province of Manabí, Ecuador. This site is primarily associated with late Valdivia ceramics, namely Phases 7 and 8, which gives it a time frame of approximately 1800–1450 BCE (Pearsall et al., 2020). The Papayita site is nestled within a diverse and fertile regional landscape characterized by coastal plains, rolling hills, and abundant water sources, an environment that would have been conducive to the establishment and sustainment of a settlement during the Valdivia period.

The recent fieldwork at Papayita has recovered a range of artifacts and ecofacts indicative of a late Valdivia occupation. A significant quantity of utilitarian ceramics associated with the terminal Phase 8 of the Valdivia sequence has been recovered. In addition, chipped and ground stone tools, as well as debitage elements were found, attesting to the daily in situ activities that, in addition to evidence of a hearth and food remains, suggest the presence of a domestic activity area. Ecofacts included bones, possibly from deer and fish, shedding light on the dietary habits and potential hunting and fishing practices of the site’s inhabitants (Fig. 2). Interestingly, marine seashells were also discovered, suggesting a broader diet and contact with coastal sources, even though Papayita is located at least 13 kilometers from the nearest shore.

The stratigraphic sequence at Papayita is of significant interest, with a base consisting of geological material layer, succeeded by a paleosol and a cultural layer (Fig. 3). The latter contains several discernible activity areas, including a hearth and small midden areas. Two separate features contained human remains associated with the Valdivia ceramics found at the site. Interestingly, the cultural layer is sealed by a significant, uninterrupted deposit of volcanic ash reaching 85 centimeters in thickness. The features discovered, as well as the artifacts’ angles of deposition suggest that this ashfall occurred while living activity at Papayita was ongoing. In other words, the site had not been abandoned at the moment the ashfall event began. The evidence of human activity after the ashfall is minimal and there is, so far, no indication of further human occupation until modern times.

METHODOLOGY

GEOCHRONOLOGY

Charcoal in volcanic ash is able to reveal ages of eruptive phases when dated properly. As there is usually enough datable carbon present, the most optimal dating methodology is $^{14}$Carbon ($^{14}$C), which may reach an age range of up to 80,000 years (Grootes, 1978; Paterne et al., 1989; Schramm et al., 2008; Dyez et al., 2014; Lewis et al., 2020). However,
the older a sample may be, the higher the error range or uncertainty of the age of the sample may become (Bronk Ramsey, 2008, 2009; Hajdas et al., 2008; Bronk Ramsey et al., 2013). At the Papayita site, we extracted several dozen charcoal samples, ensuring they remained uncontaminated by tissue or any other organic matter, in line with conventional sampling methodologies (Figueiral and Mosbrugger, 2000; O’Carroll and Mitchell, 2012; Zhu, 2014). All of the selected samples had diameters within the range of 1 to 5 mm, and, collectively, they weighed several milligrams. The age of the samples was determined using the \(^{14}\)C method at International Chemical Analysis Inc., a certified geochronology laboratory located in the USA. The methodology, accuracy and reproducibility of geochronological analyses have been presented in a variety of studies (e.g. Ardelean et al., 2020; Martinez-Pabello et al., 2021; Ryan et al., 2022). The age reported in the current study is presented as calibrated age referenced to BCE/CE (BC/AD) with age before present sometimes shown as thousands of years BP (ka, relative to CAL1950). Raw ages were calibrated using the calibration curve CalPal2007_HULU (http://www.calpalonline.de/).

**MICROSCOPY AND GEOCHEMISTRY**

Three main samples of fossil plant material or charcoal (SM-01, SM-02, SM-03), collected by the research team, were analyzed from the volcanic ash layer at Papayita (Fig. 3). These samples were small, millimeter-sized fractions of shiny black porous looking materials within the beige-grey colored volcanic ash layer. Each sample was dried in an oven (Memmert, SBN 400) at 40°C for two hours. Using a scalpel blade, several cuts of the selected grains were made and subsequently fixed to a pin for scanning electron microscopy, with an attempt to preserve the original structure as much as possible. Then, all the samples were metalized using a sputtering coater (Quorum, Q150 ES). Thus, it is possible to characterize the morphology and provenance of the plant material and its geochemical composition (González et al., 2020). For each sample, images at different magnifications were
obtained by using a Field Emission Gun Scanning Electron Microscope (TESCAN, MIRA3) operating at 10 kV. Geochemistry was performed in SEM chamber using an energy dispersive X-ray spectroscopy (EDS) detector (Bruker, X-Flash 6|30) with 123 eV resolution at Mn Kα (Toulkeridis et al., 1996; Vaca et al., 2016; Debut et al., 2021).

RESULTS AND DISCUSSION

AGE AND PROVENANCE

The geodynamic setting of Ecuador is based on the interaction between the oceanic Nazca plate with the Caribbean plate and the South American continental plate (Trentkamp et al., 2002; Mato and Toulkeridis, 2017; Montes et al., 2019; Tamay et al., 2021). The subduction of the Nazca plate is the origin of the volcanic activity, resulting in 19 recognized active continental volcanoes, with a Volcanic Explosivity Index (VEI) of up to 7 (Toulkeridis and Zach, 2017). Therefore, several volcanoes have had far-reaching recorded ash falls in the last thousands of years during the Late Pleistocene-Holocene (Toulkeridis et al., 2015; Podwojewski et al., 2022). The usual fall-out area may be around the volcano, however, due to the geographic position of the majority of the active Ecuadorian volcanoes, close and slightly south of the Equator, the predominant direction of ash-carrying clouds is towards the west to southwest, meaning towards the coastal area (Toulkeridis and Zach, 2017; Toulkeridis et al., 2022). Many pyroclastic layers as a result of far-reaching explosions have been encountered in the coastal lowlands of Ecuador and also beyond, within sedimentary deposits of the Pacific Ocean (Bowles et al., 1973; Bavlon et al., 2022). At Papayita, an ash layer with an impressive thickness of 85 centimeters was found above an underlying paleosol (Fig. 2), a fact that is striking when considering that the closest volcano with sufficient eruptive power to produce such ash volumes, Quilotoa, is around 165 km away in a direct line (Di Muro et al., 2008). Nonetheless, provenance analysis based on geochemistry analysis and mineralogy revealed that the volcano responsible for the high amounts of the expelled material must have been the Pichincha Volcanic Complex with a distance of some 225 km in a SW direction (Fig. 4; Toulkeridis et al., study in progress). The mineralogical and geochemical data coincide with the geochronological outcome of the charcoal which yielded an age of 3530 ±30 years, identical to the 3560 ±70 years BP, 3540 ±30 years BP and 3549 ±30 years BP ages of previous studies (Zeidler, 1994, 2016; Robin et al., 2008, 2010). While the previously
reported volcanic event was characterized by a limited volume of expelled material, registering a Volcanic Explosivity Index (VEI) of 4, our recent analyses of the ash layer in Papayita and nearby areas suggest a significantly greater explosivity, with a VEI of 6 (Newhall and Self, 1982; Toulkeridis et al., study in progress).

The importance of this ash layer in Papayita, as previously mentioned, is based on the fact that it covers artifacts and human remains of the Valdivian culture. Even more so, the location of the encountered human and animal bones is within the lowermost part of the ash layer, rather than under it. It is likely that the ashfall had profound consequences for a considerable percentage of the population some 3500 years ago (Ortiz-Aguilu et al., study in progress). In a similar vein to the catastrophic volcanic events at Pompeii and Herculaneum, settlements and their unassuming residents have been tragically affected by volcanic hazards (Maiuri, 1958; Sigurdsson et al., 1982; Giacomelli et al., 2003; Martin, 2020). As for the Papayita site, the data presented here is anticipated to provide valuable insights into the causes that potentially contributed or maybe even triggered the termination of the Valdivia cultural tradition in this particular region (Ortiz-Aguilu et al., study in progress).

CHARACTERIZATION OF VEGETAL MATERIAL

High-resolution three-dimensional images with surface details that allow the identification of anatomical structural characteristics and corresponding elemental chemical analysis, in petrified wood samples, have been widely used in palaeobotanical research (Penagos, 2013). Thus, it has been possible to understand life and its biological diversity in the geological or historic past (Yasuhara et al., 2017; Martinez et al., 2023). These facts allow us to establish strategies for the restoration of the natural capital (Ekins et al., 2003; Guerry et al., 2015). The recovery of knowledge about the cultural and natural environment, as observed most notably in historical sites like Pompeii and Herculaneum buried under Vesuvius volcano’s pyroclastic materials, is a prime example of this type of exploration (Bosi et al., 2011; Veal, 2014; Moser et al., 2018).

In this research, the collected material was extracted from a layer of volcanic ash (Fig. 3) in the Papayita study area. The charred wood was divided into three samples, which were sectioned and prepared for observation in a scanning electron microscope (SEM) (Vaca et al., 2016; Debut et al., 2021). In each image it was possible to determine a woody plant vascular system (Falcon-Lang, 2005; Lo Moaco and López, 2014), fibrous system, axial parenchyma and transverse parenchyma or rays (Fig. 5). This indicates two things, the first of which is that this wood underwent a carbonization process without combustion called pyrolysis (Scott and Glasspool, 2005; McParland, 2007), allowing the intact preservation of the cell wall and its middle layer, which could be homogenized (Hatipoğlu and Türk, 2009; Scott, 2010; Im et al., 2012) above a temperature greater than 300°C. Secondly,
to determine that due to the high reflectance of the charcoal samples, it can be concluded that they were plants that came into contact with hot ashes, pyroclastic deposits or basaltic lavas, where the presence of oxygen is excluded (Scott and Glasspool, 2005). Similar studies indicate that, if the wood comes into contact with volcanic ash, its carbonized material will maintain an excellent preservation of its anatomical structures, as presented in the current study (Falcon-Lang, 2005; Dufraisse, 2006; Hudspith et al., 2010), as the aforementioned coincides with the samples of microcharcoal studied, the size of which is less than 180 µm.

The three-dimensional wood fractions presented structural elements of several plant species, a woody vascular system that could be preserved through geological time (Kabukcu, 2018). These insights offer valuable information about paleoclimatology, aiding in detailed reconstructions of growth rings, their thickness, and botanical elements, alongside molecular analysis to accurately identify the species. This information aids in the understanding and in the reconstruction of the ecosystems of that time (Greguss, 1955). Furthermore, there is the presence of transverse parenchyma, in which the presence of similar growth ring structures is marked. This is a histological structure characteristic of trees that develop in cold areas or with contrasts of temperature and light, such as the Andes. It can be tentatively proposed that the fossilized wood samples are derived from trees belonging to the Gymnosperm group (Luu-Dam et al., 2023; Bufalino et al., 2023). The most recurrent anatomical characteristics of the charcoal samples were tyloïd intervacular pits or quadrangular septa, simple perforation plates, medullary rays, and woody fibers together with the tracheid-type vascular system and circular fossae or pits (Fig. 6).

There are also multiserial axial tracheids with the position of their pits with rounded or elliptical edges, in the opposite direction, their ends are more or less pointed with hollow woody tubes, and quadrangular septa (Donaldson, 1983; Fig. 7). In the cross section of its radial canals, vertical and radial quadrangular tracheids are identified. These bands of parenchyma cells extend radially perpendicular to the axial tracheids. They also present pits in the form of simple circular or elliptical holes, in homogeneous radii and absent resin canals (Pujana et al., 2014; Fig. 7). These structural characteristics have been ratified in various investigations as part of podocarp identification and classification (Patel, 1967; Correa et al., 2010; Castañeda-Posadas, 2023; Shunn and Gee, 2023).

The geochemical analysis established that in all the samples the predominant presence of elements, such as calcium, the main component of plant organic material, and silicon, which is a fundamental element in the carbonization phase. The analysis confirms that each sample corresponds to organic matter of vegetable origin, that is, carbonized wood in the form of small rocks, the appearance of which is that of woody tree trunks (Fig. 8).

**FUNGUS**

Inside a vascular bundle, a fungal structure was found, in which we observed terminal spores, intercalary spores and perfectly preserved hyphae (Fig. 9; Sutherland, 2003). The carbonized fungi considered saprophytic
remain intact due to the incomplete combustion process, thus achieving a perfect preservation of the cellular elements both in plants and in fungi and oomycetes (Wan et al., 2016). Thus, it can be seen in Fig. 9, hyphae, vesicles and spores. There are several paleomycological studies that determine this type of finding as information with good potential for the development of various types of research (Creber and Ash, 1990).

The fungi preserve their anatomical structures after the carbonization processes of the host plants, due to their chitin and sporolein content (Sutherland, 2003). Its threadlike

Figure 6. SEM photomicrographs of charred plant material, anatomical features of charcoal samples: A. Elongated and fusciform cells, tangential section, vascular system with septa without points, own to gymnosperms (VS), simple perforation plates (PP), medullary radius (RM), intervacular pits (PI), fibers (F) and tracheids (TR); B. Tangential cut: intervacular pits (PI), simple perforation plates (PP), medullary radius (RM), fibers (F), and xylem (X); C, D. Tracheids in cross section have a rounded shape, and their walls have large and less numerous pores (TR)

Figure 7. SEM photomicrographs of charred plant material. A. Tangential cut, multiserial axial tracheids (TAM) with the presence of rounded or elliptical edge punctuations (P); B. Cross section of a wooden radius, vertical tracheid (TV), radial tracheid (TR); C. Tangential cut, parenchyma, simple aerolated scores
structures are fungal hyphae in charred wood. Surely, they hosted a healthy plant or one in the process of decomposition, just when this material was dragged to the sample collection point (Dos Santos et al., 2020).

**BIOGEOGRAPHY – HABITAT**

The historical biogeography of the cloud forest in South America includes a diversity of species with different origins from both temperate and tropical zones. They have been affected by a dynamic geological environment and climate change from the Miocene to the Pleistocene, affecting the composition and distribution of several podocarp species (Dalling et al., 2011; Ornelas et al., 2019; Pandey, 2021), such as an important taxon in the northern Andean forests, during the interglacial periods 330,000 years ago (Van’t Veer et al., 2000). Coniferous or gymnosperm species are plants that grow in cold climates (Jørgensen, 2011; Ulloa Ulloa et al., 2017). Thus, the Podocarpaceae group develops in temperate and cold areas of the Andes, with an altitudinal distribution range between 1,900 and 3,800 m a.s.l. (Dodson and Gentry, 1991; Jørgensen and León-Yánez, 1999), on the floors of cloud forest and montane forest vegetation (Neill, 2012). These ecosystems surrounded the Guagua Pichincha Volcano in the past, 18,000 to 13,000 years ago. These regions registered 6 to 7°C less than the current average temperature, and where there was a greater presence of mist or cloudiness between 1,200–3,500 m a.s.l.

Currently, climate change has caused cloud forests, alteration of the hydrological cycle (Still et al., 1999; Foster, 2001; Fries et al., 2012), decrease in cloudiness and increase in rainfall, doubling of CO$_2$, as well as increase in anthropic activity like deforestation, agricultural and livestock production, population growth in buffer areas to protected areas (Toulkeridis et al., 2020). This is causing the fragmentation of ecosystems, which directly affects the distribution of plants and the change in climatic conditions (Bruijnzeel et al., 2010, 2011). Although cloud forests continue to be an area of great biodiversity (Kessler, 2022), their area is designated as a hot spot (Aguirre et al., 2021). *Podocarpus* sp. is one of the indicators or pioneer species for natural capital restoration and biodiversity conservation programs in the study area (Villamarín et al., 2009; Gardner, 2013; Bremer et al., 2019).

**CONCLUSIONS**

The development of the studied charcoal was the result of an incomplete combustion or pyrolysis process, above 300°C, which determined the conservation of the anatomical structures of the wood fragments as a microorganism within a vascular bundle.

Histological structural elements such as woody vascular bundles – xylem, quadrangular tracheids, with pits in the shape of circular or elliptical holes, transverse parenchyma and absence of resin canals, characteristics that correspond to a species of woody tree from the group of Gymnosperms, of the family Podocarp. In the paleobotany area, there were not enough samples to define the species, since the specific identification of the wood was realized by comparing the anatomical structure of the aforementioned genus in forests that currently exist.
Climate change has caused the alteration of the hydrological cycle, decreased cloudiness, increased rainfall, doubling of CO₂, in the cloud forests and montane forests of the buffer zone of the Guagua Pichincha volcano. In addition, the increase in anthropic activity, such as deforestation, agricultural and livestock production, demographic growth in the area, has generated fragmentation in the ecosystems, affecting the distribution of plants and the change in climate conditions.

The species belonging to the Podocarp family inhabit temperate and cold areas of the Andes with an altitudinal distribution range between 1900 and 3800 m a.s.l., that is to say that the volcanic activity of the Guagua Pichincha Volcano transported Podocarpus wood from a cloud forest of the high Andean zone to the Papayita site located in a coastal environment near the sea level where it was preserved. The vast ashfall and the subsequent charcoal-laden ash layer, coupled with the ensuing environmental disturbances, were probable key contributors to the ending of human occupation at the Papayita settlement and may have played a part in the termination or final metamorphosis of the Valdivia cultural tradition in this region ~3500 years ago.

ADDITIONAL INFORMATION

CONFlict of interest. The authors have declared that no competing interests exist.

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