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Reconstruction of Fire History Using 'Dry' Sediments, An Approach for Microcharcoal Studies from the Sierras Pampeanas, NW Argentina

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ABSTRACT
A new accessible and easy method for studying past fire events using 'dry sediments' is presented. The method was developed for analysing samples from 17 locations in the Ambato Valley, NW Argentina, to study fire regimes in the area. Based on earlier researchers' methods, guidelines to use microcharcoals in 'dry' sediments as a palaeoenvironmental proxy are provided. The procedures outlined include sampling/extraction in the field, preparations of the samples, laboratory analysis and quantification of the samples. The new approach was useful to meet the objectives and answer the questions initially proposed in our research about past fire regimes affecting the past Aguada society.

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Fire history; microcharcoal; pedoanthracology; methods; fire regime; fire ecology

Introduction

In this paper we synthesise and discuss briefly earlier methods developed to study past fire regimes and their relevance in the field of pedoanthracology and microcharcoal studies. We present a new method for reconstructing fire events using 'dry' sediments from fluvial contexts. Our case study was conducted in the Sierras Pampeanas, the Ambato Valley, Catamarca Province, in Argentina, an area which is affected by regular forest fires. We wanted to develop a new easy, inexpensive method which could be use in our study area to help us understand past fire events which had impacted on the Aguada Culture in the Ambato Valley and to be able to analyse the possible relationship between past forest fires and their abandonment (Lindskoug 2010, 2014, 2016a, 2016b; Lindskoug and Marconetto 2014; Marconetto et al. 2014, 2015; Marconetto and Lindskoug 2015) as well as be applicable to other locations which lack appropriate lacustrine sediments with a cyclical deposition normally analysed for microcharcoal to study past fire events.

The disappearance of the Aguada culture occurred around 900–1000AD and was first proposed by Alberto Rex González (González 1979, 1998; González and Pérez Gollán 1972) who argued for violence and ecological deterioration of the environment as possible causes. Archaeological data, accumulated over years of excavation in the area, have provided a robust body of evidence, with several contexts interpreted as scenarios of abandonment that affected multiple archaeological sites associated with this society in the Ambato Valley. Several of these large sites, including Piedras Blancas, Martínez 2, and La Iglesia de Los Indios (sometimes referred to as La Rinconada), showed signs of fire associated with the abandonment. Recent fieldwork suggests that these events may have been simultaneous (or almost simultaneous), a perspective strengthened by ^{14}C dating (Laguens 2006; Marconetto et al. 2014). More recent discussions of the abandonment of the area have been presented by Gordillo (2013), Gordillo and Leiton (2015), Gordillo and Vindrola-Padró (2017) and Lindskoug (2016a, 2016b). Our studies (Marconetto 2009, 2010) have also found evidence of a drought at the time of site abandonment of the valley, which further inspired us to investigate the possible relationship between abandonment and forest fires. However, our aim here is to present our new approach to study past fire regimes using microcharcoal used during this investigation.

Microcharcoal research has had great influence on the field of vegetation history as many researchers count microcharcoal particles when counting pollen on microscope slides in order to obtain information about past fire regimes with most of the studies of this type have been carried out on lake sediments (e.g. Hawthorne and Mitchell 2016; Whitlock and Larsen 2001). Such studies are usually not specifically directed at studying fire regimes in the past, as is the aim here. Therefore we decided to experiment with microcharcoal extracted from 'dry' sediments to see if it was possible obtain information on past fire regimes and to resolve questions related to the disappearance of the Aguada culture. To accomplish our aim, it was necessary to develop this new method to study the

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past fire events from 'dry' sediments because no lacustrine deposits exist in the study area which could be used employing more traditional methods. Since there are not any other methods using dry sediments from similar environments it is not possible to compare this new method with older ones, however we will discuss the advantage of this new method here.

The field of microcharcoal studies (or pedoanthracology) is based on the identification and dating of microscopic charcoal recovered from soils. Taxonomic identification is possible in larger sized charcoal fragments, but in many cases only counting is performed (Abdoun, Thinon, and Alifriqui 2000; Talon, Carcaillet, and Thinon 1998). The first person to define pedoanthracology was Thinon (1978), who set up a framework and methodology for this relatively recent discipline. Later, Carcaillet and Thinon (1996) developed techniques for studying 'dry' sediments in mountainous areas. However, there is no universal method for microcharcoal analysis, nor for the counting of particles, and various authors have developed their own techniques in this field, for example see discussions in Patterson, Edwards, and Maguire (1987). One advantage of pedoanthracology is that samples are not biased by the selection of certain species by people.

A range of studies of past fire regimes have focussed on microcharcoal extracted from different types of sediments. Many researchers are working with very different perspectives on charcoal and a range of different approaches exist in different disciplines, including archaeology, geology, biology, chemistry and physics. This means that some researchers might not be aware of each other's work, which is sometimes published in specialised books and journals (see Whitlock and Larsen (2001); Conedera et al. (2009) and Scott (2010) for the range of studies).

Charcoal occurs in the natural environment often as a result of wildfires, volcanic activity or as a result of human activities. Not all charcoal is the result of unintentional fires, since it can be produced to be used for domestic or industrial processes as well. Pedoanthracology also offers a complementary method for the palaeoenvironmental reconstruction of vegetation to that based on pollen records (Di Pasquale et al. 2008, 2010; Thinon 1978). Microcharcoal can also be used to understand past climate change and vegetation dynamics, since small changes in climate may alter the fire regimes. Other researchers working on the same issues have made important contributions (e.g. Scott and Damblon 2010; Scott, Moore, and Brayshay 2000; Turner et al. 2010; Turner, Roberts, and Jones 2008). In the case of Argentina, most studies related to this field have been carried out in Patagonia. For example, Huber and Markgraf (2003) studied the impact of European colonisation on natural fire regimes and vegetation dynamics in southern Patagonia. Other studies focused on the relationship between human impact and climatic change using microcharcoal evidence (e.g. Haberzettl et al. 2006; Heusser 1987, 1994, 1999, 2003; Huber, Markgraf, and Schäbitz 2004; Iglesias, Markgraf, and Whitlock 2016; Kitzberger and Veblen 1999, 2003; Kitzberger, Veblen, and Villalba 1997; Markgraf and Anderson 1994; Markgraf, Whitlock, and Haberle 2007; Sottile 2008; Veblen et al. 1999; Whitlock et al. 2006; Whitlock, Moreno, and Bartlein 2007). In this study the focus will be on microcharcoal and how it is used in the reconstruction of fire regimes in a part of NW Argentina, where no studies have been undertaken.

Methodological issues concerning microcharcoal and the reconstruction of fire events

The abundance of microcharcoal can vary on a local scale and can differ in two sites sampled less than 100 m from each other. It is therefore important to first select good sampling points in all locations. Local variations must be considered and each sampling point must be thoroughly recorded and described. The selection of sampling areas outside of archaeological sites should be guided by the recent geological history of the region in order to find the best areas, especially those that operated as natural sediment traps. Another issue to consider is the possible mixing of sediment layers due to natural processes, such as bioturbation, which can be frequent.

In terms of methodology, the most common method for reconstructing fire histories has been the counting of charcoal particles by palynologists on pollen slides (Rhodes 1998; Whitlock and Millspaugh 1996). In this way it is possible to reconstruct palaeoenvironmental changes based upon both pollen and microcharcoal records from the same sample. Turner (2007) evaluated several different methods used and found several problems. Charcoal is normally divided into three classes: microcharcoal (<180 μm, mesocharcoal (180–1 mm) and macrocharcoal (>1 mm). Elements larger than 180 μm can be eliminated in the cleaning process in pollen preparation. The process is rigorous and involves several chemical and mechanical treatments (Rhodes 1998, 114). These procedures are used to 'clean' the pollen sample of both inorganic and organic materials contained in the sediment matrix. Whereas pollen grains are highly resistant, charcoal is not and it is likely that 'cleaning' results in fragmentation and thereby an overestimation of charcoal when counted (Clark 1984 cited in Turner 2007, 36). The process of chemical cleaning is also both costly and time-consuming.

The size of the charcoal fragments can be used to determine whether the fire was local or regional (Patterson, Edwards, and Maguire 1987). 'Large fragments' generally indicate a nearby source and 'small fragments' are normally considered to have dispersed over longer distances by the wind. Experimental studies have been performed to evaluate the production of microcharcoal after wildfires and its behaviour in terms of its transport by wind and water (e.g. Blackford 2000; Clark 1988a; Higuera et al. 2007; Nichols et al. 2000; Patterson, Edwards, and Maguire 1987; Peters and Higuera 2007). Clark (1988a) developed models and formulas for charcoal transport with have several limitations that relate to the processes of deposition and fragmentation of the charcoal. For example, Nichols et al. (2000) have shown that water transports larger charcoal particles a greater distance than smaller particles and this must be taken into consideration if the source of the fire is to be determined.

As yet, there is no universal methodology to study microcharcoal. The methods normally used include 1) petrographic thin sectioning (Clark 1988b); 2) the Oregon sieving technique (Millspaugh and Whitlock 1995); 3) bleaching and filtering (Rhodes 1996, 1998); 4) density separation (Clark 1984), which according to Turner (2007, 44) has not been sufficiently refined yet; 5) density separation and bleaching (developed by Turner, Kelly, and Roberts 2008 on the basis of Clark's (1984) technique) and, 6) a technique employed by Carcaillet and Thinon (1996) that involves the digging of large trenches to obtain samples for flotation which are later dated by AMS. The material recovered with this approach is usually larger in size than in the other methods mentioned, focusing more on mesocharcoal and macrocharcoal. The first three methods are the ones most commonly used to study fire regimes along with the sixth technique developed by Carcaillet and Thinon (1996).

Another issue is related to the quantification or counting of particles. Three different standard methods are normally used: 1) absolute abundance measurement involves the counting of all particles, regardless of their size (Patterson, Edwards, and Maguire 1987); 2) point counting developed by Clark (1982) and is based on the number of hits using an eyepiece graticule, usually 11 points. The particles that are not in the field of view are ignored. This method was developed to allow a rapid measurement of charcoal content; and 3) area measurement method developed by Waddington (1969) and is widely used. This method is based on a gridded graticule and quantifies the percentage area occupied by microcharcoal. Each charcoal particle is measured and assigned to a size class to provide additional information about the source of the fire and transport of the particles etc. This method is time-consuming but since it involves the measurement of the particles it can also provide important transport information. This method also has many disadvantages, as discussed by Turner (2007) and others. Several researchers disagree about the relevance of the size classes for microscopic charcoal (Tinner and Hu 2003), especially because of the fragility of microcharcoal (Marquer 2010; Turner 2007).

Another method for estimating microcharcoal particles is image software analysis, first performed by Horn, Horn, and Byrne (1992). This method is not yet sufficiently developed and there are several problems with counting since the software cannot discriminate between charcoal remains and other black particles found in the microscope images. Not all black fragments are charcoal and other materials that are easily 'mixed' include weathered pyrite, marcasite, and biotite (Rhodes 1998, 114). There are also some methods that are based on subjective estimates, involving an estimation of the charcoal content of a sample on either a five- or seven-point scale or on a percentage basis. This approach is highly subjective and not commonly used today, although it can be used when large amounts of charcoal particles are found in the samples. The two most commonly used counting methods are absolute particle abundance and the size class method. Consequently, this was taken into consideration when developing the methods of extraction and sample preparation here.

Another problem in the identification of plant microcharcoal is that when observed under the microscope it can sometimes be confused with other black-coloured elements. Some researchers count all black elements in the samples, which in many cases may be another material such as black mineral content. If plant anatomy is observed this does not pose a problem, although this sometimes depends on the type of sediment. For example, some microcharcoal particles may be 'dirty' and their anatomy can be obscured in heavy clay making identification difficult. Remains of insects can also be confused with microcharcoal, so it is also necessary to perform an analysis of the matrix to determine whether the presence of dark minerals can interfere with the count.

Study area the Ambato Valley

The Ambato Valley is located in the province of Catamarca in northwest Argentina. The valley runs north to south with the Ambato or Manchao mountain range to the west (4,050 m asl) and the Sierras Graciana-Balcosna range to the east (1,850 m asl). The valley's northern limit is the Altos de Singuil highlands and the southern boundary is the Catamarca Valley. The Río de Los Puestos emerges from the Altos de Singuil and runs north to south across the fluvial plain that forms the floor of the valley, with the river's name later changing to Río del Valle.

The valley forms part of the geological province called the Northwest Sierras Pampeanas, an area characterised by narrow valleys alternating with high mountain ranges and forests. The geological formations in the valley include El Portezuelo/Ancasti, Concepción and Coneta, along with alluvial deposits on the valley floor. The geological components include the basement rock, mainly banded gneisses, migmatites and schist outcrops, which are often intruded by pegmatites and tonalite-granodiorite bodies. Quaternary sediments along with small relict areas of Tertiary sedimentary rock fill the inter-mountain valley (Blasco et al. 1994). The principal components of the El Portezuelo/Ancasti formation are constituted by the metamorphic basement formed in the Early Precambrian-Palaeozoic period and include granular gneisses and migmatitic banded gneisses, gneissic schists, or mica-rich gneisses, composed of quartz, plagioclase, biotite, sillimanite and/or cordierite (Blasco et al. 1994, 19). The quaternary sediments correspond to the Concepción (Pleistocene) and Coneta (Holocene) formations. The sediments grouped in the first level of the piedmont are composed of fanglomerates and correspond to the Concepción formation. The second level of the piedmont is the Coneta formation, which is composed of fanglomerates, sands, and silts (Blasco et al. 1994).

The region forms part of both the 'Monte' and 'Chaqueña' phytogeographical provinces (Cabrera 1976). The valley is characterised by a warm continental climate, with annual precipitation ranging from 500–800 mm, mainly occurring as localised summer rains (November-March). It is noteworthy that the area represents a border zone with other phytogeographical provinces with different characteristics. To the northwest, it borders with the *Prepuna* and *Monte* phytogeographical provinces (Cabrera 1976), and towards the northeast it adjoins the extreme southern end of the *Yungas*, the (Selva) Tucumano-Boliviana jungle/rainforest of the Amazonian Dominion.

The characteristic vegetation of the valley is arranged in 'belts' or 'bands', each one presenting a particular structure and composition. The first band corresponds to forest features *(bosque serrano)*, followed by a band of shrubs and grasses. At higher altitudes the woody taxa disappear, replaced by an almost pure grassland (*pastizal de altura*). The altitude ranges occupied by each band vary as a function of latitude and longitude and also in response to microclimatic conditions, especially in relation to the orientation of the slopes (Morláns and Guichón 1995). The western and eastern slopes of the valley have different vegetation because of the differences of precipitation that the two mountain ranges receive. Since the Ambato Valley is located in a transition zone between two different phytogeographic regions it contains species from both zones. However, there is a clear difference between the vegetation growing on the western slopes of the valley and on the eastern slopes.

De la Orden and Quiroga (1997) created a physiographic classification that divides the valley into several areas, although the landscape has changed since this classification was created. Today, agricultural activities have largely transformed the valley, especially the southern part because of the large-scale cultivation of walnuts. This has accelerated the deforestation of the native species in the valley, a process that has been aided by the use of heavy machinery in other agricultural activities. This means that the vegetation surveys of the 1990s are no longer valid in some parts of the valley and in many places the native vegetation has been totally replaced, with only small relic patches surviving. The use of heavy machinery and ploughing to clear new agricultural land has also damaged or totally destroyed some archaeological sites in the valley.

Materials and methods

We decided to use microcharcoal for several reasons: 1) we detected high amounts of microcharcoal in soil samples retrieved from fluvial contexts in our study area; 2) the area today is regularly affected by wild land fires; 3) microcharcoal is indicative of past fire events on both a local and regional scale and we work in a large valley system which gave us the possibility to analyse past fire regimes and changes in settlement pattern on different scales; 4) the present vegetation in the area leaves high amounts of microcharcoal after wildland fires; and since Marconetto (2009, 2010) have shown there are no significant changes in the vegetation since the first occupation of the area. We chose not focus on mesocharcoal and macrocharcoal here which can be used in anthracological studies for taxonomic identification as we have several papers focusing on these charcoal fractions (Espósito and Marconetto 2008; Lindskoug and Mors 2010; Marconetto 2002, 2003/2005, 2005, 2007, 2008; Marconetto and Gordillo 2008; Marconetto and Lindskoug 2015; Marconetto and Maffera 2016; Marconetto and Mors 2010).

The aim of this work was to develop a way to extract microcharcoal from 'dry' sediments. The first step was to select a test station location in the field (Profile 1), sample it and recover the microcharcoal in order to evaluate the method and refine the laboratory techniques (see below). After evaluating these samples, the extraction procedures in the field were refined and also the methods used in the laboratory. We also obtained reference samples after recent forest fires in Córdoba province to assist with the identification process. Finally, during a field campaign in 2010, 17 stations were sampled using a sediment corer and these are the basis for this study (Figure 1). As there is no single universal technique to extract, prepare or count the samples, we conducted an experimental study to test our method that could be applied more widely and too avoid using numerous different methods which complicates the comparison of results between studies.

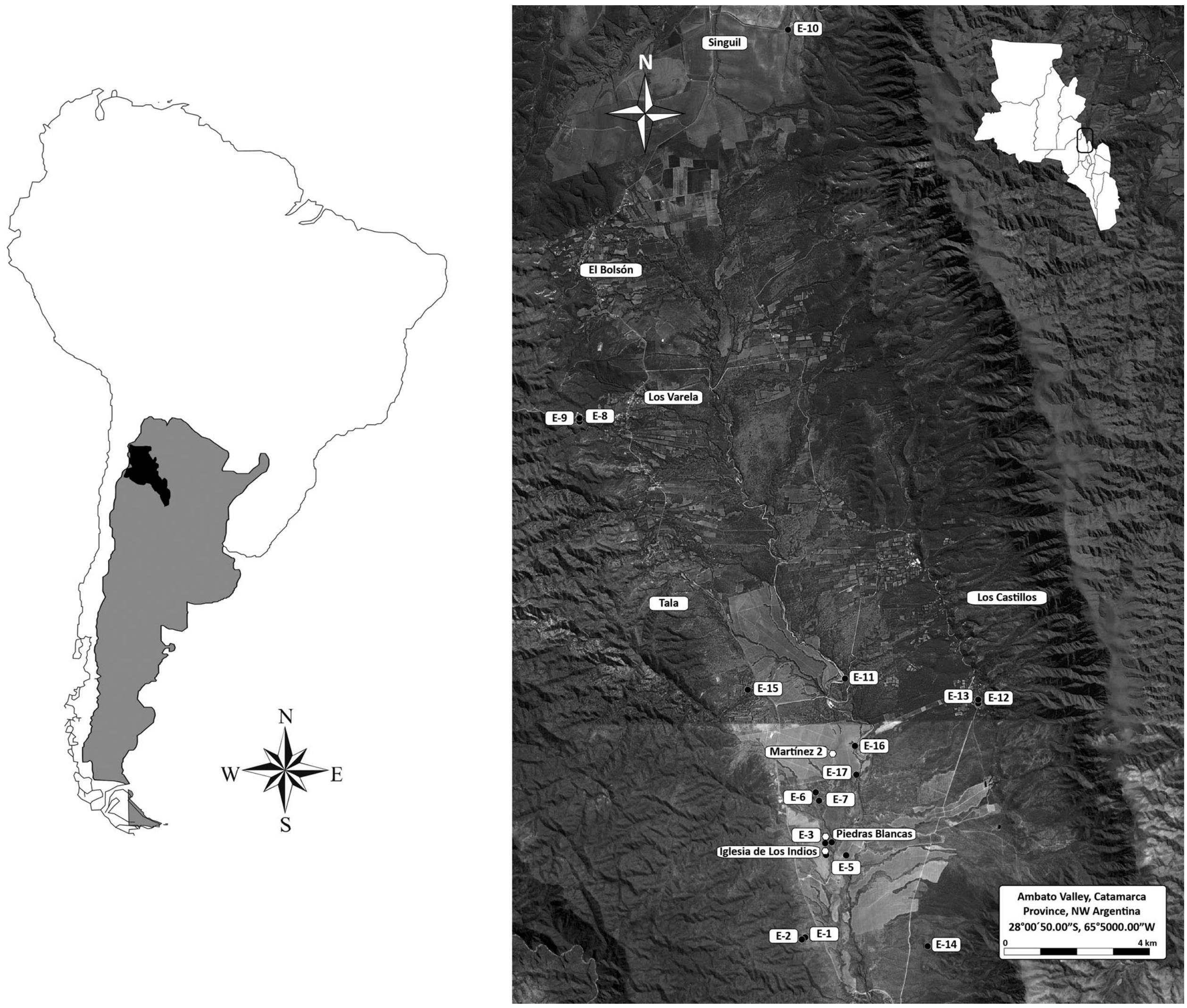

Figure 1. Map of the study area showing the 17 stations sampled in the Ambato Valley with key archaeological sites affected by fire.

Profiles extracted in the field

In 2008, the sediment samples from Profile 1 were collected in a gully close to the river edge with a trowel and stored in small plastic containers (Figure 2). The profile section was first cleaned and then the samples were collected from the surface level down to avoid contamination of the soil samples. In total 21 soil samples were collected, each 5 cm thick from the surface down to 105 cm, and a brief description of the samples was made in the field (see Table 7.1 in Lindskoug 2016b). One larger sample was collected from the same section between 110 and 130 cm as it contained large amounts of organic material. It was radiocarbon dated to 2,370 ± 60 ^{14}C years BP at LATyR (Table 2).

17 stations were sampled from north to south, located in different areas of the Ambato Valley (Figure 1) and some reference samples were also collected from the surface. The stations were carefully selected with the assumption that they served as sediment traps for the transported microcharcoal, depending upon natural factors such as wind and rainfall conditions. Other criteria for selection were based upon location and inclination of the terrain, ranging from the valley floor to river terraces, and included present and past river channels (Table 1). Some stations were also sampled on the hillsides in prehistoric agricultural terraces, taken close to prehistoric settlements and inside the archaeological structures. The altitude of the sampling locations ranged between 1,061 m asl and 1,259 m asl. An extensive wildland fire occurred in the area eight months before the sampling took place (Figure 3), but since almost no rain had fallen since, signs of the fire were still clearly visible and large amounts of microcharcoal were present on the surface revealing the fire affected areas. We also categorised the samples extracted to different areas: 1) areas directly affected by the fire, 2) areas located close to the area affected by the fire, around 300 m from the fire source, and 3) areas located further away from zones affected by the fire. By interviewing local people more information was obtained about the extent and intensity of the fire that took place in December 2009 (Figure 3). The stations directly affected by the fire were stations 1–4, 6, 14, and 15.

Figure 2. Photo of Profile 1, sampled in October 2008.

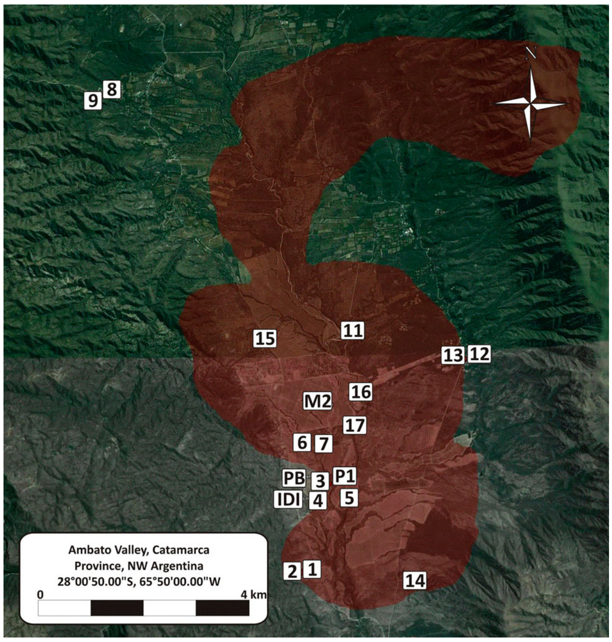

Figure 3. The area on the floor of the Ambato Valley affected by fire in December 2009.

Sediment samples were extracted with a corer, which was a mechanical model with reinforced extensions, a cylindrical point (20 mm × 400 mm), and a maximum length of 3 m (Figure 4). The sediment cores were usually divided into sections of 10 cm, although at times subdivisions were made after a preliminary inspection demonstrated interesting changes or features in the core (see Lindskoug 2016b, for a full description of each station). The minimum depth of coring was 40 cm and the maximum was 235 cm. Differences in core length were related to the fact that some areas were too stony, or the soils were too hard or shallow, for example, the sediments in the agricultural terraces were not very deep. The cores from each station were subdivided into 3–26 samples, with most ranging from 8 to 17. In total, 226 samples were collected from the 17 stations. The samples were stored in ziplock plastic bags and labelled to indicate the station, sample number and geographic position. A preliminary classification of the sediments was performed in the field based upon colour, soil morphology, particle size and sorting using a commercial grain size chart (INTEQ P/N 18834). Local vegetation, the terrain, and other factors that could affect the sample were also noted (Table 1).

Laboratory procedure

The sediments were left to dry at room temperature and were then analysed using Motic BA200 microscope in transmitted light with 40x, 100x, 200x, and 400x magnifications. Photos of the different

Table 1. Geomorphology and vegetation for the stations in the Ambato Valley.

Station	Geomorphology	Vegetation after De la Orden and Quiroga (1997)	Altitude (m asl)
Station 1	Piedmont- limit of the third terrace of the Los Puestos River	Grassland with Shrubbery (P3P)	1087
Station 2	Piedmont- limit of the third terrace of the Los Puestos River	Grassland with Shrubbery (P3P)	1084
Station 3	Fluvial floodplain- The second terrace of the Los Puestos River	Gallery-Forest Flatland (LLBg)	1078
Station 4	Fluvial floodplain- The second terrace of the Los Puestos River	Gallery-Forest Flatland (LLBg)	1076
Station 5	Fluvial floodplain- The second terrace of the Los Puestos River	Forest Flatland (LLB)	1061
Station 6	Fluvial floodplain- The second terrace of the Los Puestos River	Gallery-Forest Flatland (LLBg)	1087
Station 7	Fluvial floodplain- The second terrace of the Los Puestos River	Gallery-Forest Flatland (LLBg)	1083
Station 8	Piedmont	Forest with Shubbery (P3MPA)	1257
Station 9	Piedmont	Forest with Shubbery (P3MPA)	1259
Station 10	Second terrace of the Los Puestos River almost piedmont	Shrubbery (P4A)	1226
Station 11	Fluvial floodplain- edge of the terrace between the fist and second terrace of the Los Puestos River	Gallery-Forest Flatland (LLBg)	1106
Station 12	Piedmont	Open Forest (P2B)	1224
Station 13	Piedmont	Open Forest (P2B)	1225
Station 14	Piedmont	Open Forest (P2B)	1112
Station 15	Third terrace of the Los Puestos River almost piedmont	Grassland with Shrubbery (P3P)	1126
Station 16	Fluvial floodplain- The second terrace of the Los Puestos River	Gallery-Forest Flatland (LLBg)	1089
Station 17	Fluvial floodplain- The second terrace of the Los Puestos River	Gallery-Forest Flatland (LLBg)	1085
Profile 1	Fluvial floodplain- The second terrace of the Los Puestos River	Gallery-Forest Flatland (LLBg)	1073

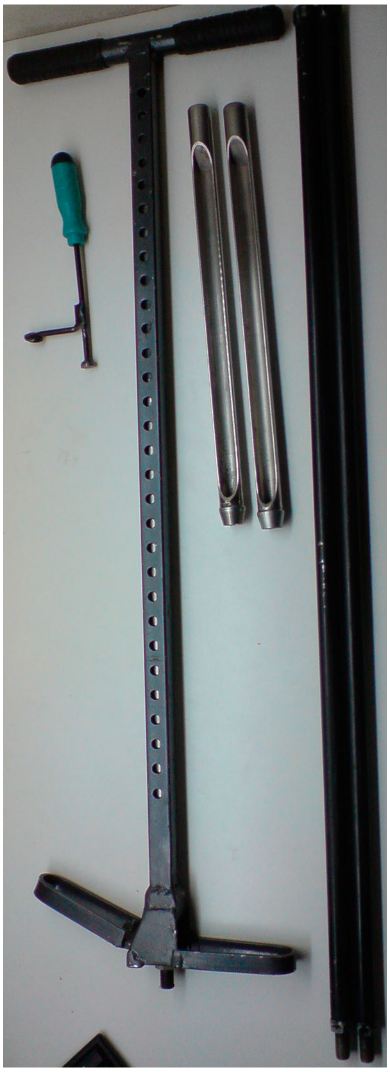

Figure 4. The sediment corer used for extraction in the field.

features found were also taken with a Motic digital camera (Moticam 1000, 1.3 megapixel). The procedure for the preparation of the samples was the following: a small standard amount (0,0025 g) of soil/sediment was placed on a microscope slide, then five drops of sewing machine oil were added. The sample was left for approximately 30 s to disperse the oil, at which point a glass cover slip (20 × 20 mm, 0.13–0.17 mm thick) was placed on top. Sewing machine oil was used because it is a highly refined mineral oil resistant to heat. Mineral oils are petroleum based and much more stable than other types. They also support the heat released by the light of the microscope well. The samples were then sealed with clear nail varnish to produce a semi-permanent preparation that could be used for several analyses. After the sample had been left to dry it was ready to be analysed (see also Lindskoug 2010, 2015).

This method allowed us to observe microcharcoal to around 30 μm. An area of 4 cm^2 was scanned for each sample. In the first analysis, two categories were used to evaluate samples: presence and absence. The features identified in the samples were phytoliths, black vegetal structures, black minerals, microcharcoal, fibres, elongated silica structures, and indeterminate black elements (Figures 5 and 6). The category 'black vegetal structure' was used when the cellular structure of the microcharcoal was clearly visible. This was done to differentiate the microcharcoal in which the cellular structure was not visible but could be identified because of its form, colour and shape, especially notable edges (Figure 7). Turner, Kelly, and Roberts (2008) argue that accurate identification of microcharcoal can be problematic for several reasons. For example, there are black elements in sediments that can resemble charcoal, such as pyrite, dark plant fragments and insect cuticles. They also state that the actual identification is subjective and strongly depends upon the experience of the analyst. Therefore the same identification criteria as Turner, Kelly, and Roberts (2008) was applied in order to improve the identification of the microcharcoal. These diagnostic criteria are 1) jet black colour, 2) angular, straight edges, 3) straight but fuzzy edges, 4) blue hue, 5) the presence of cellular structure (see Figure 1 in Turner, Kelly, and Roberts 2008). If a combination of these criteria were applicable the elements were classified as microcharcoal or black vegetal structure, depending upon the visibility of the cellular structure. The first step of the analysis evaluated the presence and absence in the samples of the features listed above. The next step in the research was the counting and quantification of the microcharcoal identified in the samples.

Chemical treatment of the samples was avoided due to concern that cleaning them would lead to mechanical damage and an overestimation of charcoal abundance and also the lack of the proper laboratory equipment to use chemicals was taken into consideration. Therefore it was decided to process more samples without chemical cleaning. Because of this, elements classified as black vegetal structure (carbonised elements with a recognisable cellular structure) were considered to be safer indicators of fire than the elements classified as microcharcoal. Of course, if elements with a black vegetal structure were recorded this was also indicative of microcharcoal in the samples. See Appendix section for the experimental study leading to our new technique.

The first stage of the analysis revealed that there were problems in identifying the microcharcoal from biotite (black mica) in some samples. In fact, all of the samples in this study contained black particles, and that these did not necessarily corresponded to traces of plant charcoal. To address this, experimentation with flotation in distilled water and centrifugation of the samples using filter paper to extract the microcharcoal was done. This approach proved to be time consuming so it was decided to use the first preparation technique. To solve the

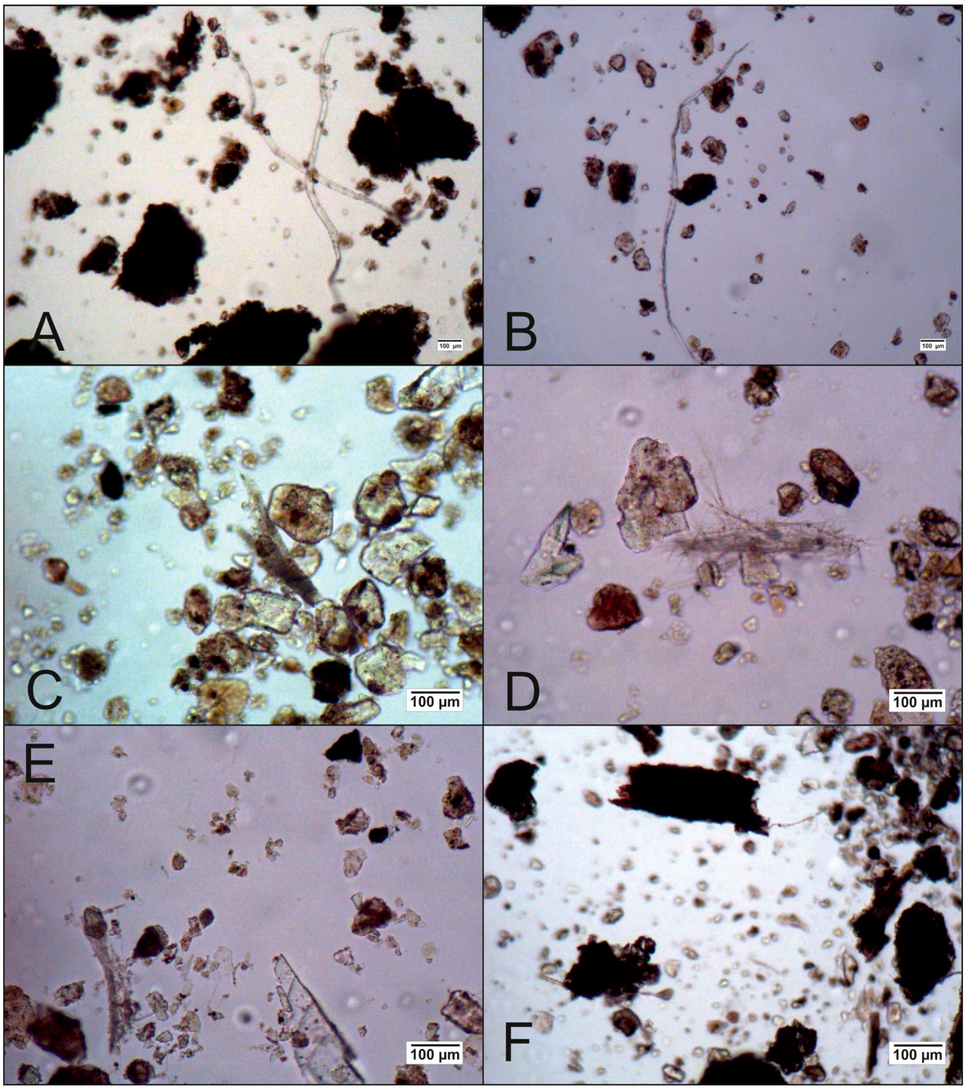

Figure 5. Photos of some features identified in the samples: A) fibres from Station 17, sample 8 (100x); B) fibres from Station 17, sample 11 (100x); C) articulated phytoliths in plant tissue, Station 2, sample 6 (200x); D) raphides, Station 2, sample 6 (200x); E) articulated phytoliths in plant tissue and microcharcoal, Station 2, sample 6 (200x); F) microcharcoal, Station 10, sample 1 (100x).

problem of large amounts of biotite (black mica) in the sample, we used polarised light, as it allows black mica to be distinguished from charcoal because it shines under polarised light. This technique was applied to several samples that contained large quantities of microcharcoal and also for samples presenting a medium or low count, in order to evaluate whether the elements being quantified were actually microcharcoal particles. The extraction processes already had identified samples with a high content of black mica and this helped to select the samples for analysis. Many black mica particles were found, although using Turner's criteria none of these would have been classified as microcharcoal. It was impossible to make a total count using polarised light because the samples were too dark to identify the charcoal particles, but this method helped to establish the robustness of the method and to ensure the charcoal content in the samples was not overestimated due to a high mica content (Figure 8).

Quantification of the microcharcoal content

After the initial presence/absence study it was necessary to quantify the microcharcoal. We opted for Absolute Abundance Measurement method (Patterson, Edwards, and Maguire 1987), which involves counting all of the microcharcoal particles found in the 215 samples from the 17 stations. We used the same samples for the quantification as used in the presence/absence study but excluded the three reference samples.

Radiocarbon dating of the samples

The last step in the process was to select samples for AMS dating in order to better correlate the fire signals in terms of chronology and sedimentary position (depth) of the samples (Table 2 and Figure 9). The samples were dated at the NSF-Arizona AMS Laboratory. We decided to date samples which had high

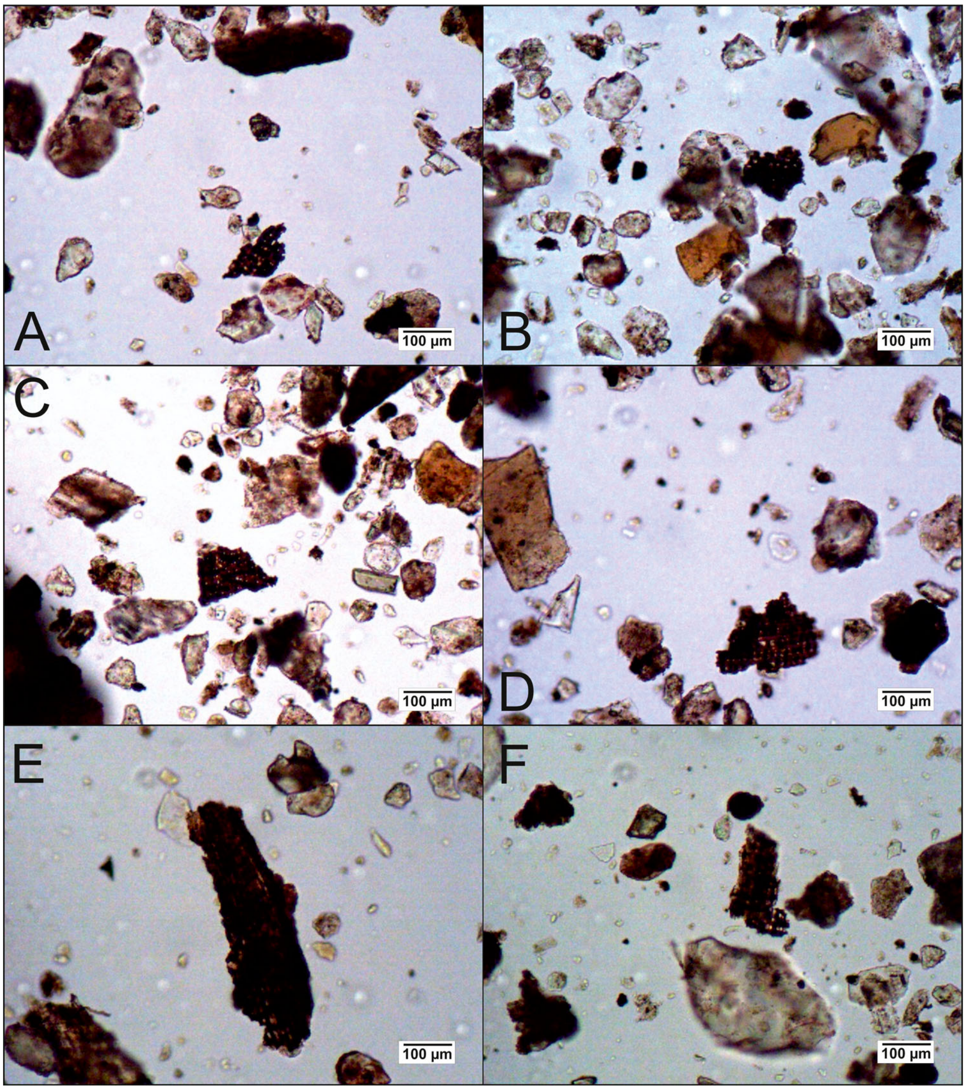

Figure 6. Photos of microcharcoal particles with black vegetal (cellular) structure from Station 16 (200 x). A) Sample 2; B) Sample 1; C) Sample 3; D) Sample 2; E) Sample 5; F) Sample 4.

quantity microcharcoal with recognisable black vegetal structure, mainly because these fragments were larger than the rest. Ten samples were dated for 17 stations sampled (Lindskoug 2016b; Lindskoug and Marconetto 2014). The presence/absence study provided guidance for selection of the samples to be dated. The quantification of the microcharcoal in the samples must be performed in order to find out where the strongest or most continuous fire signals are found, in order to obtain better information about the past wildfire regimes in the area.

Results

The results of the analysis of particles with black vegetal structure are presented in Figure 9, indicating presence/

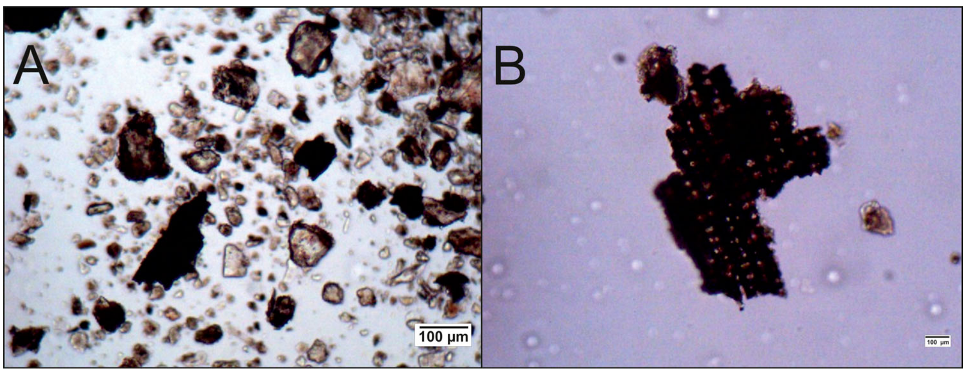

Figure 7. Photos of microcharcoal (A) and black vegetal (cellular) structure (B). A) Station 10, sample 1 (100 x); B) Station 5, sample 8 (200 x).

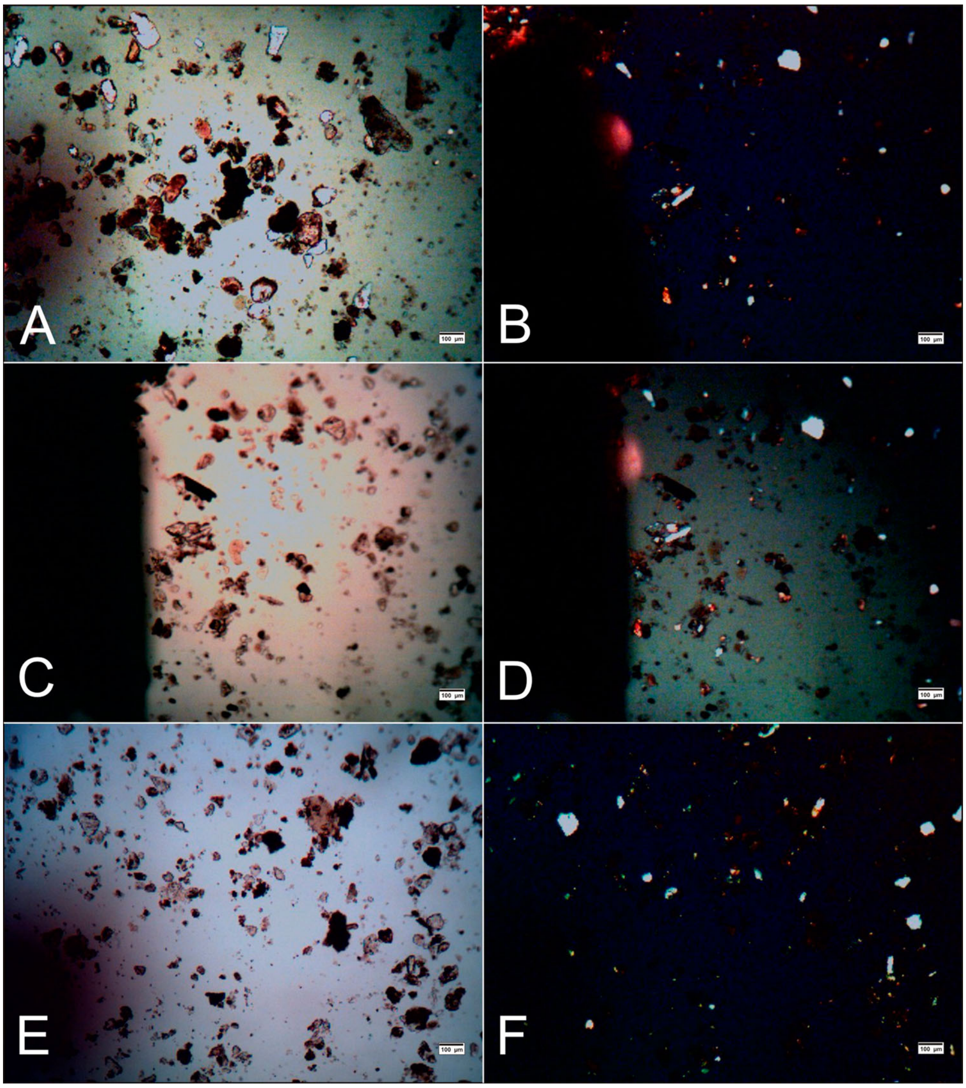

Figure 8. Photos testing polarised light from Station 8, sample 1 (100 x). A) B) C) D) E) F).

absence in each level/sample from each station as well as the number of samples per station (see also Lindskoug 2014; Lindskoug and Marconetto 2014). The full results of all microcharcoal frequencies are shown in Figure 10. All of the stations show variability in fire frequencies at different depths, with all of the stations having large quantities of microcharcoal in the first section except for Stations 5, 7 and 12. Many stations also have large quantities of microcharcoal (>100 particles) in the oldest two samples, except for Stations 1 and 2. A total of 23,487 microcharcoal particles were identified in the 215 samples analysed, with an average of 1,382 per station. Station 13 had one of the lowest counts with 467 particles identified (Table 3 and Figure 10) whilst station 10 was the highest with 2,509 particles (see Table 3). A low count was obtained from Station 5 at a

Table 2. AMS radiocarbon dates from stations in the Ambato Valley from NSF-Arizona AMS Laboratory.

^{14}C date number	Laboratory code	Station	Depth (cm)	Material	Δ 13 C	F	^{14}C age B.P.
1	AA94243	Station 3	90–100	Sediment	−19.5	0.7420 ± 0.0082	2.397 ± 88
2	AA94244	Station 3	225–235	Sediment	−16.7	0.6707 ± 0.0078	3.209 ± 94
3	AA94245	Station 10	135–145	Sediment	−23.4	10.257 ± 0.0042	post-bomb±
4	AA94246	Station 17	60–75	Sediment	−16.9	0.6999 ± 0.0032	2.867 ± 37
5	AA94247	Station 17	115–120	Sediment	−16.4	0.5672 ± 0.0030	4.554 ± 42
6	AA97979	Station 3	10–20	Sediment	−23.6	1.0048 ± 0.0030	post-bomb±
7	AA97980	Station 3	40–50	Sediment	−24.0	1.0523 ± 0.0031	post-bomb ±
8	AA97981	Station 5	50–60	Sediment	−19.6	0.9717 ± 0.0030	231 ± 25
9	AA97982	Station 6	30–40	Sediment	−19.5	0.8817 ± 0.0077	1.012 ± 70
10	AA97983	Station 17	20–30	Sediment	−21.0	10611 ± 0.0046	post-bomb ±
Conventional radiocarbon date from LATYR							
	LP-2211	Profile 1	~110–130	Sediment with organic material	−24 ‰ ± 2		2.370 ± 60 years B.P.

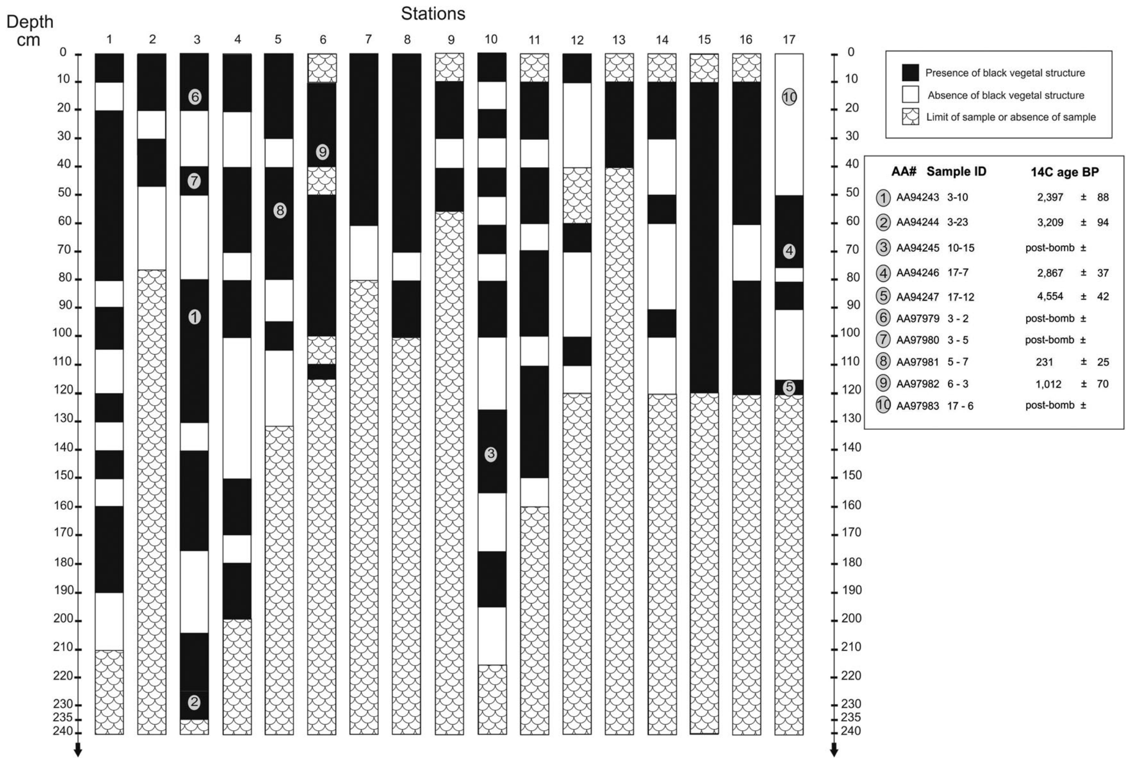

Figure 9. Presence/absence of elements with black vegetal structure, i.e. carbonised vegetal matter, in the stations sampled in the Ambato Valley, with AMS radiocarbon dates.

depth of 80–92 cm and this station also had the lowest average count of all of the stations with 59 microcharcoal particles per sample (Table 4).

Large differences were found for the minimum and maximum counts: 18 particles was the lowest in Station 5, and 105 the highest in Station 16 (10–20 cm). The average minimum count for all samples is 57 microcharcoal particles. The maximum value, 386 particles, was found in the upper sample (0–10 cm) of Station 8. Station 8 also has the highest average microcharcoal content per sample (169 particles). Station 7 had the smallest maximum content of microcharcoal, with 141 particles at a depth of 60–70 cm. The average maximum count for all stations was 213 particles per sample and on average there are 117 microcharcoal particles per sample, if all samples are

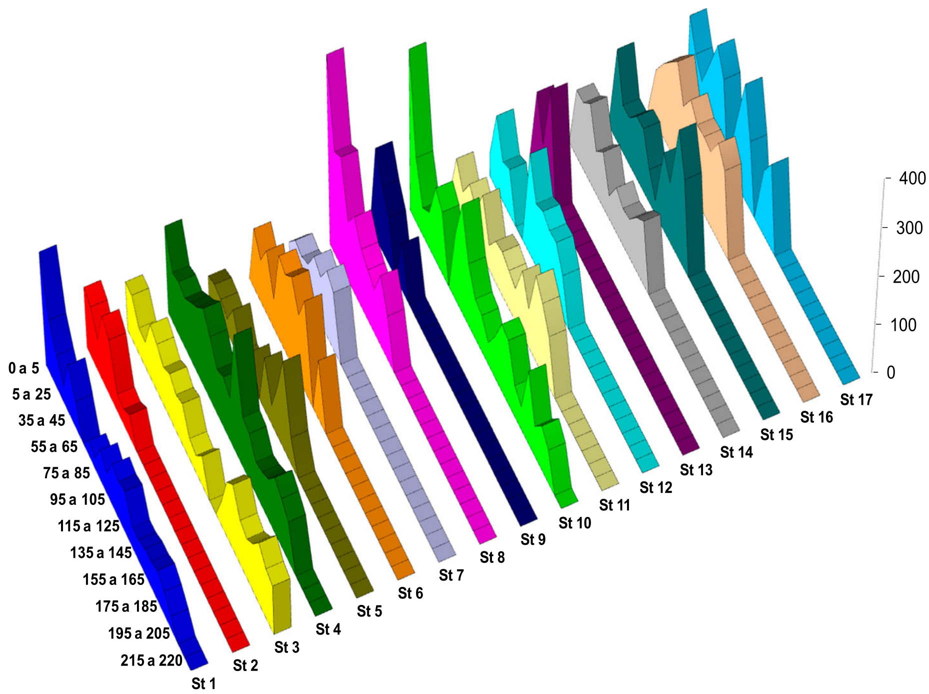

Figure 10. Results of all microcharcoal counts from all 17 stations sampled in the Ambato Valley. The scale to the right indicates microcharcoal quantities and the Y-axis indicates the depth of the samples in cm.

Table 3. Charcoal quantities and frequencies from all stations.

Station	Total number of samples	Minimum microcharcoal	Maximum microcharcoal	Total sum microcharcoal	Average microcharcoal per sample
Station 1	21	25	235	1555	71
Station 2	8	35	147	705	88
Station 3	23	39	147	2101	91
Station 4	20	72	254	2449	122
Station 5	16	18	190	940	59
Station 6	9	92	196	1364	152
Station 7	8	40	141	729	91
Station 8	10	92	386	1688	169
Station 9	4	99	189	523	131
Station 10	22	45	328	2509	114
Station 11	15	50	175	1451	97
Station 12	10	48	203	1176	127
Station 13	3	83	220	467	156
Station 14	11	46	157	1136	103
Station 15	11	27	251	1401	127
Station 16	11	105	199	1717	156
Station 17	12	57	197	1576	131
Total sum	214			23487	
Average	13	57	213	1382	117
Minimum	3	18	141	467	59
Maximum	23	105	386	2509	169

taken into account. Further discussion and analysis of the results can be found in (Lindskoug 2016b; Lindskoug and Marconetto 2014).

Discussion

The stations sampled on the fluvial floodplain in the central part of the valley, which are associated with archaeological settlements (Station 3, 4, 5, 6, 7, 16, and 17), recorded several peaks in microcharcoal throughout the sequences. Large concentrations and peaks in counts are also found around 110–120 cm, except at Station 3. However, materials at these depths are probably too early to reflect the human settlement in the valley, considering the depth of the AMS dates obtained until now. Instead, these must reflect an early increase in fire regimes in the valley. Based upon the results obtained it can be suggested that the study area has been regularly affected by wildfires since at least 4,500 BP, which is the age of the oldest AMS date obtained from the samples. All stations recorded a peak in microcharcoal content around 40–45 cm, but the total count differs among stations (Figure 10). An AMS date from Station 6 (for the 30–40 cm depth) provided an age of 1012 ± 70 ^{14}C BP, suggesting that this depth corresponds to the abandonment of the valley by the Aguada culture. There are generally low microcharcoal counts thereafter in all these stations. The reason for this is not totally clear but this period might correspond to a phase or interval with increasing moisture and biomass expansion and a later period with higher aridity, fire frequency and biomass burning. Several stations that were also affected by the last fire in December 2009 had high frequencies of microcharcoal in their surface layer. It must also be noted that sedimentation rates may differ between different locations in the valley, and that therefore a depth of 30 cm in two different stations does not necessarily correspond to the same age. However, if stations were sampled in nearby locations and have

Table 4. Data from profile 1.

Sample number	Depth (cm)	Sediment layer	Black elements	Black vegetal structure	Black minerals	Microcharcoal
MS-P1-1	0–5	1	x	–	x	–
MS-P1-2	5–10	2	x	–	x	x
MS-P1-3	10–15	3	x	–	x	x
MS-P1-4	15–20	3	x	–	x	–
MS-P1-5	20–25	3	x	–	x	–
MS-P1-6	25–30	3	x	–	x	x
MS-P1-7	30–35	4	x	–	x	x
MS-P1-8	35–40	4	x	?	x	?
MS-P1-9	40–45	5	x	–	x	–
MS-P1-10	45–50	5	x	?	x	?
MS-P1-11	50–55	5	x	?	x	?
MS-P1-12	55–60	6	x	?	x	?
MS-P1-13	60–65	7	x	?	x	?
MS-P1-14	65–70	8	x	?	x	?
MS-P1-15	70–75	9	x	–	x	–
MS-P1-16	75–80	9	x	–	x	–
MS-P1-17	80–85	10	x	–	x	x
MS-P1-18	85–90	10	x	–	x	–
MS-P1-19	90–95	11	x	–	x	x
MS-P1-20	95–100	12	x	x	x	x
MS-P1-21	100–105	13	x	x	x	x

similar characteristics it can be assumed that the sedimentation rate has been rather similar and may correspond to a similar chronology.

The stations sampled in the terrace system show fluctuations throughout the entire section. Stations 1 and 2, sampled in the southern part of the valley in the piedmont zone, correspond to the Grassland with Shrubs (P3P) vegetation unit. As the stations sampled are close to each other it can be assumed that they have a similar sedimentation rate. The top sample from both stations is similar, with high values, followed by a decrease and then a peak at a depth of 30–40 cm, then another increase at a greater depth. However, no radiocarbon dates have yet been obtained from any sample taken in the agricultural terrace system or in the piedmont area, which complicates the possible chronological correlation of the depths in this area or in this kind of agricultural terrace structure but shows that fire regimes can be reconstructed from these kind of archaeological contexts.

Some variability occurs between the stations. Stations 12, 13, and 14 were also grouped together based upon their many similarities (same vegetation unit and located in the southern piedmont zone of the valley). However, the stations differ a great deal between each other and not many similarities were found in the microcharcoal frequencies. This might reflect the fact that all three stations were sampled in different kind of structures: an agricultural terrace, a settlement unit, and a possible archaeological water reservoir. If Station 12 is compared to Stations 1 and 2, which were both sampled in the southern piedmont area of the valley, some similarities between them can be found. Some peaks and declines in the top sections seem to correspond with the depths of the samples obtained at all three stations.

All of these stations sampled in this study do however show varying frequencies of microcharcoal particles through their depths, which demonstrates that the fire regimes have been shifting over time in the valley and also that some areas might have been more affected than others by the prehistoric settlements and human activity. The results are discussed in full in Lindskoug (2016b) and it is not the aim to discuss them here.

Recently, we have also initiated palaeoenvironmental studies concerning the past vegetation and fire dynamics in the study area, using ecological modelling (Burry et al. 2017; Marconetto et al. 2015). Using Hindcasting Ecosystems Model (HEMO), we have model past ecosystem dynamics using remote sensing in the same area ranging from 442 to 1998 CE (Marconetto et al. 2015). This model uses Normalised Difference Vegetation Indexes (NDVI) to make hindcasts about past vegetation patterns in the Late Holocene. Further studies on the past fire events using ecological modelling have demonstrated that this model can help us further understand the variations in the past fire regimes and to interpret different archaeological contexts related to fire events, especially when it is unviable to radiocarbon date all soil samples extracted in the field, as discussed in this paper.

This approach has also been used successfully by Di Lello (2015) to study fire regimes in the locality of Yacanto de Calamuchita, Córdoba province in central Argentina, and been proven useful in this case. Both the Ambato Valley and the Calamuchita Valley forms part of the Sierras Pampeanas and they are subject to the same orographic system. Two profiles were extracted and studied with our approach, indicating that fire have been an integrated part of the natural environment before the introduction of *Pinus* in the area in middle of the twentieth century. We hope that this approach will be used be others interested in past fire regimes in other parts of the world.

Conclusions

Advantages of the new approach

Whilst several approaches to reconstruct fire histories through microcharcoal already exist, the approach outlined here allowed us to develop a new tool to study dry sediment. It has number of advantages to obtain information on past fire regimes. It is an easy and accessible method, it minimises mechanical and chemical fragmentation and avoid over estimation of the microcharcoal and it opens up the study of fire histories into areas which have not been investigated before because of lack of lake sediments or other suitable archives. There is the possibility of identifying different fire ‘signs’ in the archaeological record.

This new approach allowed us to identify microcharcoal and other elements in the samples, then make quantitative studies using the microcharcoal identified, compare the samples representing the different units sampled in the field, and radiocarbon date different events Another important contribution with our new approach is that the microcharcoals are not further fragmented due to chemical treatment or mechanical damage as the samples are not cleaned which would in many cases lead to an overestimation of charcoal fragments in the samples. Laboratory procedures are also easy and accessible and do not need expensive chemicals or laboratory equipment.

Contributions to solve problems posed related to our archaeological research

Our new approach to study past fire regimes using microcharcoal from dry sediments has enabled us to analyse past fire history and examine the relationship with settlements patterns in the Ambato Valley, especially the abandonment of the area (Lindskoug 2016a, 2016b).

Further our new approach has enable us to has shown that natural fires have been a common occurrence in the area and that fire is an integral part of the ecosystem of the Ambato Valley. Microcharcoal has been found in all of the sites sampled, although with differences in terms of presence/absence in different layers (Lindskoug 2016a, 2016b; Lindskoug and Marconetto 2014). The presence of microcharcoal in various layers indicates that there have been periods with a higher frequency of fires. Whether this is related to human activity, natural processes, or perhaps a mixture of both remains to be determined. But based upon the results obtained it can be concluded that the study area has been regularly affected by wildfires since at least 4,500 BP, which is the age of the oldest AMS date obtained from the samples. However, since microcharcoal has been found in samples even deeper than the oldest one dated, it is likely that this environment was regularly affected by fires for a longer time, even before the first known evidence of human occupation appears in the Valley.

These studies have also provided new insights into the palaeoenvironmental conditions and context in the valley and has deepened the understanding of the area's geological and environmental history, developing a deeper vision of the history of the Ambato Valley.

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Appendix section

The development of the new method

We did several tests with our microcharcoal samples from Profile 1 to evaluate different methods to prepare the samples in a laboratory environment (Table 4). The first step in our experimentation was by analysing the samples without any processing, using a Kyowa Unilux-12 binocular microscope with magnification up to 400 x, and using both transmitted and reflected light. The preparation of the samples was simple: a small amount of sediment was mounted on a microscope slide, and tested mounting with distilled water, sewing machine oil, and immersion oil. The sample was left for approximately 1 min to disperse the oil, at which point a glass cover slip (20 × 20 mm, 0.13–0.17 mm thick) was placed on top. The samples were then sealed with clear nail varnish to produce a semipermanent sample that could be used for several analyses. After the sample had been left to dry it was ready to be analysed. The best and easiest mounting medium proved to be the sewing machine oil. Continued experimenting with the samples to see if it was possible to obtain better results/visibility and clean out other black elements, for example black minerals such as biotite, which were quite abundant in some samples. Filtration was tried with filter paper (Munktell diameter 110 mm, density 84 g/m2) as well as flotation in Petri dishes both with and without the use of filter paper, but these processes were very time-consuming.

The most advanced test with flotation was done at the Environmental Archaeology Laboratory (MAL) at Umeå University in Sweden during a short research stay. A standard amount of sediment was poured into a test tube (13 × 100 mm). It was then filled to two-thirds full with distilled water. A Vortex-Genie 2 shaker was used to speed up the flotation process for the microcharcoal. Then a pipette (50 µm) was used to extract the sample from the test tube. However, the last step with the pipette was abandoned because a large amount of the material got stuck inside the pipette. The sample was then mounted on a slide with a glass cover slip and studied under the microscope. Our experiment with Profile 1 helped to select and develop the techniques later applied in this study, such as the preparation methods and the identification of different features in the samples.