Archaeological Science

An Introduction

Edited by

Michael P. Richards Simon Fraser University

Kate Britton University of Aberdeen



Geoarchaeology Panagiotis Karkanas

The application of geoscience-based methods to archaeological sites to understand site formation processes and use.

1 INTRODUCTION

In its broadest definition, geoarchaeology is the study of the archaeological record using any geoscience-based technique, method, concept, or knowledge (Rapp and Hill 2006). However, since archaeometry is a well-defined field focusing on the application of physical sciences to archeological prospecting, dating, and provenance (Waters 1992), it could be proposed that geoarchaeology has a more narrow definition, actually closer to the original coining of the term (Renfrew 1976) and to its modern main application. In this approach, geoarchaeology is the discipline that studies site stratigraphy and site formation processes, and the interaction of human and nature in shaping the landscape (Butzer 1982; French 2003; Goldberg and Macphail 2006; Waters 1992;). The history of this approach goes back several hundred years, as can be seen in the 1863 monograph of Sir Charles Lyell: Geological Evidences of the Antiquity of Man. However, it was not until 1976 that Colin Renfrew introduced and defined the term "geoarchaeology" in the preface of an edited volume by Davidson and Shackley (1976). Indeed, Renfrew (1976) defined precisely what should be the main concern of geoarchaeology, concisely summed up by Goldberg and Macphail (2006: 3): "geoarchaeology provides the ultimate context of all aspects of archaeology from understanding the position of a site in a landscape setting to a comprehension of the context of individual finds and features."

That being so, the scale of practicing geoarchaeology varies from a regional perspective scale to that of a single site. Although a combination of offsite and

on-site study will help with understanding the position of a site in the larger geomorphic system (Butzer 2008), there is usually a dichotomy between geoarchaeologists dealing with site formation processes and geoarchaeologists focusing on landscape studies. This does not imply that there are no integral perspectives or that there are no sites (e.g., early hominin sites) in which a mixed approach is always followed (see Ashley et al. 2009 and discussion in Butzer 2008; Quade et al. 2004). A possible distinction does exist and refers to whether we are looking for the context of the site as a whole or not. On-site geoarchaeology requires a range of expertise dictated by the infinitive repertoire of anthropogenic site-settlement activities, natural sedimentary facies that are not normally studied in larger scale projects (e.g., rain or sheetwash, ponding and very small-scale mass wasting and sediment gravity-flows) and site-specific post-depositional alterations and disturbances (e.g., trampling, dung decay, wood ash alteration). On the other hand, landscape geoarchaeology is a more straightforward application of geomorphology and environmental sciences. Nevertheless, both approaches are interdisciplinary and share the same ultimate goal, that is, the study of archaeological context. In addition, site-specific formation processes are not independent of the larger geomorphic system; therefore a good on-site geoarchaeological study needs to integrate the site processes into the surrounding landscape. Thus it appears that the aforementioned dichotomy never exists in reality but it can be theoretically accepted because it serves the specific objectives of archaeology.

The following summarises the main aspects of landscape and on-site geoarchaeology and provides some examples of their application.

2 LANDSCAPE GEOARCHAEOLOGY

The landscape approach dominates geoarchaeological research. It is mostly of regional perspective scale, aiming a) to reconstruct the landscape for understanding site locations, distributions and spatial changes, b) to recognise how natural and human-induced processes alter the landscape and c) to identify intentional manipulation of the environment (forest clearance, cultivation, manuring, irrigation systems, dams, land recreations, etc.).

The first objective is commonly associated with regional archaeological surveys, which attempt to locate unidentified sites and to trace changes in settlement pattern through time, often in relation to the distribution of natural resources. Geoarchaeological study aims to facilitate sampling strategies, prioritise survey regions, and provide an environmental framework for survey data interpretation (Wells 2001). In addition, a major goal is the prediction of the location of buried sites (Ferring 2001; Gladfelter 1985; Hassan 1985). In a first stage, the geoarchaeological survey requires the recognition of landform formations. In a later stage, the reconstruction of the palaeolandscape will show the relation of the settlements and land-use practices with the landscape during specific periods. According to Wells (2001), three distinct kinds of geomorphological data are needed to accomplish this goal: stability, chronology, and palaeoenvironment. The determination of the stability (stable, depositional, erosional, or mixed) of the geomorphological surfaces will show which artefacts have been reworked and explain why some surfaces are sterile (due to erosion or sedimentary burial). The age of the geomorphic surface will determine what parts of the landscape were extant during any particular period. Finally, the reconstruction of the palaeolandscape during a particular period will provide the ultimate context of the artefacts and sites. A good example is the geoarchaeology of the Great Plains in North America where the reconstruction of its geomorphic history was used to understand the presence and temporal distribution of Paleoindian sites and eventually contribute to the issue of peopling of the New World (Mandel 1992; Mandel 2000). Geomorphic, chronostratigraphic, and soil-stratigraphic data were used to predict buried sites for each cultural period in the river basins. It was shown that geological processes have affected the archaeological record by either removing or burying sites that date to certain cultural periods not presented in the area.

Furthermore, landscape geoarchaeology is concerned with setting a particular archaeological site in its immediate environment. It is interested in integrating the whole site into major landscape features and understanding what circumstances governed its location, and defined land-use practices, but also in investigating what affected its subsequent preservation over the longer term (e.g., Draut et al. 2008). This approach tries to put the archaeological site as a whole in its regional context and can be considered in its broader sense as site formation processes (Goldberg and Macphail 2006). For example, the geoarchaeological study of the alluviation history of the floodplain surrounding the medieval city of Alzira (Spain) illuminated its constructional and settlement history, such as expansion and shrink, and abandonment and occupation (Butzer et al. 1983). The geoarchaeological reconstruction of the past landscape and environment at the Neolithic Çatalhöyük (Turkey) was directly linked to the excavated on-site evidence for subsistence. It was shown that the bulk of the cereal agriculture was not carried out in the immediate vicinity of the site as the area was flooded each spring and thus would

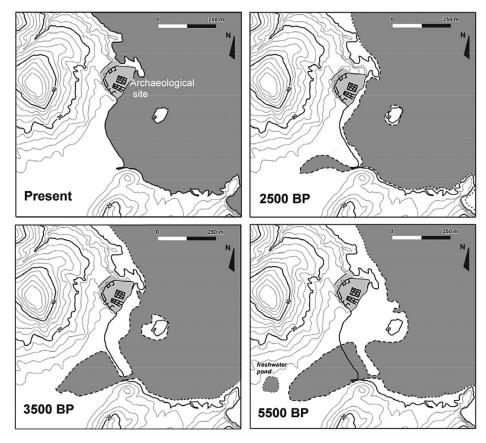


FIGURE 13.1 Palaeogeographical reconstruction of the coastal area in the Bay of Palamari, Skyros Island, Greece. Note that the extent of Palamari archaeological site during antiquity is not known in the presently submerged area (see Pavlopoulos et al. 2010).

have damaged any autumn-sown cereal crops (Roberts and Rosen 2009). Another example comes from the reconstruction of the palaeoenvironment and coastal evolution at the Bronze Age Palamari fortification in Skyros Island, Greece (Pavlopoulos et al. 2010). The flourishing of the settlement is probably related to the existence of a sheltered and protected lagoon connected to the sea between about 4000 and 1500 BC. The decline of Palamari might be related to geomorphological and environmental changes that rendered the embayment a restricted body of water (Figure 13.1).

The second objective of landscape geoarchaeology includes the documenting of all landform changes, such as tectonic and sea-level changes; aeolian and periglacial

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processes; erosional and alluvial events; and recognizing all the effects of humans in altering the environment. The ultimate goal is to differentiate natural from humaninduced changes, find their interrelationships and understand how people managed such changes (Frederick 2001; French 2003). Intensive geoarchaeological research in the past decades has made considerable progress in assessing the impact of human activities in a naturally changing environment. The Mediterranean environment, for example, has witnessed several stages of erosion and formation of alluvial fill. Vita-Finzi (1969) in his pioneering classic investigation of Mediterranean alluviation introduced a general model of aggradation restricted to two well-defined time periods. Later, Wagstaff (1981) showed that the Holocene alluviation history of Greece was much more complicated than Vita-Finzi thought. Van Andel et al. (1986), in an intensive geoarcheological study of the Argolid peninsula, Peloponnese, Greece, suggested that the Pleistocene alluvial sediments could be a climatic result but the Holocene series was essentially the result of human impact. Van Andel and his team expanded their research further north to Thessaly, Greece, where they documented earlier erosional and depositional phases attributed to the clearance of the land by rising Neolithic farming populations (Van Andel and Runnels 1995). Although several other scholars have contributed to expanding the record of alluvial and erosion episodes in Greece, what is interesting is the ongoing debate on the causes of these episodes as to whether they are human induced, natural, or multi-causal (see discussion in Bintliff 2002 and Butzer 2005). At this point, close links between regional geoarchaeological research and basic research in climatic changes, tectonics, and geomorphology (to name a few) should be underlined. Although both types of research have their own agendas, in the last decades, geoarchaeological research has gained much from palaeoclimatology, for example; conversely it has also contributed to some degree in studying past climatic changes (see Karkanas et al. 2008; Karkanas et al. 2015).

The third objective, although of regional importance, is often based on smallerscale studies. It is actually concerned with direct human impacts on the environment through a number of activities. Medium-scale land recreations are often encountered in urban archaeology. By using a series of cores, Ammerman (1996) was able to reconstruct the relief of the area of the ancient Agora at Athens. He demonstrated that a major transformation of the landscape was necessitated at the end of the sixth century BC to build the well-known complex of monuments and relocate the drainage system. In the same vein, the detailed sedimentological and stratigraphic work of Huckleberry (1995; Huckleberry 1999) on the prehistoric Hohokam agriculturalists (Arizona) that practised canal irrigation provided a detailed record of palaeofloods and channel changes. These were partly responsible for the changes in their settlement pattern.

Soil evidence identifying forest and woodland clearance, cultivation, and manuring have been found in several cases. Soil micromorphological features, charcoal analysis, pollen, phytoliths, and chemical changes are all used to provide evidence of soil disturbances associated with agricultural practices (Courty et al. 1989: 126–137; Davidson and Carter 1998; Goldberg and Macphail 2006: 193–210; Macphail et al. 1990). Disposal of urban waste in arable lands has also been successfully recognised (Davidson et al. 2006). In some cases, extensive soil modifications and deterioration of vast areas have been attributed to human impact (Macphail et al. 1987). The use of agricultural terracing is very well known in the Mediterranean area. Detailed stratigraphic, soil, and sediment analysis showed interesting implications for the preservation of the archaeological record; early agricultural landscapes and soils; and past land use, as well as for interpretation of local records of Holocene erosion and valley alluviation (Krahtopoulou and Frederick 2008).

Most of the studies related to landscape geoarchaeology employ traditional soil science, geological, and geomorphological methods. These include recognition and analysis of geomorphological features, which establish a sequence of landforms (morphostratigraphy), for instance an alluvial terrace sequence (Ferring 2001; Mandel 1992). Another method is the identification of alluvial facies bounded by discontinuities (allostratigraphic units). The latter connote a significant change in the depositional regime, caused by climatic changes; tectonic activity; or base level changes (Ferring 2001). Furthermore, identification of sequences of buried soils (Holliday 2004; Mandel and Bettis III 2001) could be a fundamental tool for stratigraphic correlations (pedostratigraphy) and dating the sediments (geochronology) will provide the necessary time framework. In addition, more detailed soil studies (pH, organic matter, cation exchange capacity, chemical analysis, etc.) and analysis of soil formation processes (soil micromorphology) can be used in landscape and climatic reconstruction (Angelucci et al. 2007; Macphail 1986). A variety of sedimentological analytical approaches (sedimentary structures, grain-size analysis, mineralogy, chemical analysis, magnetic parameters, etc.) are used to characterise the depositional environment and determine the source of the sediments (Woodward et al. 2001). In addition, palaeoenvironmental indices such as micro-charcoal, pollen, phytoliths, ostracods, and diatoms are employed for reconstructing the palaeolandscape (Glais et al. 2017; Pavlopoulos et al. 2006; Sadori et al. 2004).

3 ON-SITE GEOARCHAEOLOGY

Site geoarchaeology is more neglected, although it is now becoming of increasing importance in geoarchaeological investigations (Butzer 1981; Goldberg et al. 2007; Goldberg et al. 2009; Karkanas et al. 2007; Karkanas et al. 2015; Karkanas and Goldberg 2019; Karkanas and Van de Moortel 2014; Macphail et al. 1997; Macphail et al. 2017; Matthews et al. 1996; Mallol 2006; Mentzer et al. 2017; Milek and Roberts 2013; Miller et al. 2013; Shahack-Gross et al. 2005; Shillito and Matthews 2013; Weiner et al. 1993). It is concerned with the deposits found at a site, and with what people have left behind. This microscale approach is focused on the formation processes that built the site and actually deals with archaeological sediments *sensu stricto* (Goldberg and Macphail 2006).

In contrast to landscape geoarchaeology, the study of occupational deposits demands a nontraditional approach and analyses. Site microstratigraphy and microfacies analysis using microscopic techniques (Courty et al. 1989; Courty 2001) are combined with traditional bulk sedimentological methods (i.e., granulometry, bulk chemistry, see also above) focused on deciphering the special nature of anthropogenic deposits. In particular, such an approach involves micromorphology, the study of intact sediments and soils at a microscopic scale (Courty et al. 1989), and mineralogical, microchemical, or physicochemical analysis of soils and sediments using instrumental techniques (Goldberg and Macphail 2006: 335–367). By using this combination of techniques it is possible to unravel specific human activities, identify the use of a space and to understand the depositional context of all archaeological remains.

As opposed to architectural sites (e.g., urban centers), in non-constructed sites (e.g., palaeolithic sites) cultural deposits mainly consist of burnt and other organic remains. In these sites, the study of the microstatigraphy and microstructure of cultural deposits can unravel specific burning activities and use of space, such as *in situ* burning and dumping areas, or activities related to cleaning and modifying living sectors (Goldberg 2003; Goldberg et al. 2009; Karkanas and Goldberg 2019: 171–197; Meignen et al. 2007; Miller et al. 2013). Post-depositional alterations tend to obliterate combustion features, either by ash recrystallisation (Karkanas et al. 2007) or chemical alterations (Karkanas et al. 2000; Karkanas et al. 2002; Schiegl et al. 1996; Weiner et al. 1993). These post-depositional processes have serious implications for the preservation of all archaeological remains, since each type of archaeological material is stable under certain chemical conditions and can dissolve when found in a different geochemical regime (Karkanas et al. 2000). Organic matter, phytoliths, bone, chert or flint, wood ash, and sometimes even charcoal could be

destroyed, thus impoverishing the archaeological record and even leading to erroneous interpretations. In some cases there are good indications, direct or indirect, of the past presence or absence of these materials (for a review see Karkanas 2010).

Natural sedimentary processes dominate at non-constructed sites. The superimposition of natural and cultural processes in the same depositional unit produces unique sedimentary structures and contents that cannot easily be identified by the naked eye. To disentangle these processes someone has to detect fine-scale grading, crude sorting, and orientation of particles, sometimes in mm-thick layers inside predominately anthropogenic deposits. All of the above are the result of a particular natural depositional regime that provides the basic framework for interpreting the context of the archaeological remains (Courty et al. 2001; Goldberg et al. 2007; Karkanas 2001; Karkanas et al. 2015; Karkanas and Goldberg 2019: 21-98; Macphail and McAvoy 2008; Mallol et al. 2011). Nonetheless, the type of processes can also be linked with the spatial patterning of the cultural remains at the specific site (Lenoble et al. 2008). Climatic signals have also been detected in archaeological sedimentary sequences. In particular, open caves and rockshelters have been proved to be very promising in revealing climatic changes (Courty and Vallverdu 2001; Karkanas et al. 2008; Karkanas et al. 2015; Woodward and Goldberg 2001) because caves act as sedimentary traps. The study of the cave sedimentary sequences has employed both traditional sedimentological analyses (Butzer 1981) and micromorphology (Goldberg et al. 2009; Karkanas et al. 2015; Karkanas and Goldberg 2013; Mallol et al. 2011; Miller et al. 2013; Shahack-Gross et al. 2014).

The Paleoindian site of Wilson-Leonard is an example of using on-site data to understand both the relation of the site to the changing palaeoenvironment (actually in the realm of landscape geoarchaeology), and the identification of human behaviors at the site. At this site, sedimentary facies and microfacies analyses and their vertical and lateral variations were used to reconstruct the site settlement and sediment history. Wilson-Leonard was occupied when a fluvial channel was abandoned by avulsion. Fluvial sediment continued to accumulate on the site from the nearby river but gradually became less important and covered the site only during major floods. Concomitantly the occupation of the site increased, along with the use of numerous burnt rock ovens and the production of fire-related organic matter that was mixed with colluvium material derived from the slopes behind the site (Goldberg and Macpail 2006: 33–37, and references therein).

One of the best examples of site geoarchaeology is the study of stabling activities in southern Europe where occupational deposits interchange in time and space, with stabling remains as a result of the complex (almost idiosyncratic) nature of human activities (Golberg and Macphail 2006). On the basis of differences in the nature of the components and the microstructure and arrangement of dung remains, it was possible to differentiate between animal species and possible food sources. Furthermore, specific human practices were identified, such as dung burning for clearing purposes and the construction of floors (Karkanas 2006; Macphail et al. 1997; Boschian and Montagnari-Kokelj 2000).

Site formation processes in urban and other architectural sites are even more complicated (Karkanas and Goldberg 2019: 199-221). Natural sedimentary features are not well expressed. These sites are dominated by small-scale gravity-based processes, rain wash, and aeolian activities (e.g., disintegration and gradual collapse of houses). Microscopic water-laid surface crusts, well-sorted wind-laid sands, and graded bedding in puddles are good indications of unroofed areas (Matthews and Postage 1994; Matthews 1995). Moreover, geoarchaeological studies of occupational sequences in tells have provided valuable information on social behavior, change and organisation (Karkanas and Efstratiou 2009; Karkanas and Van de Moortel 2014; Matthews 1995; Matthews et al. 1996). A set of sediment characteristics is related to different maintenance and discard practices and specific activities (Karkanas and Goldberg 2019: 138-148; Karkanas and Van de Moortel 2014; Matthews 1995). Evidence of dumping, trampling, sweeping, and food preparation, storage, and cooking were identified in several cases as the micro-content and, particularly, the fabric of the sediment are indicative of the different mechanisms involved in their formation (Courty et al. 1989; Ge et al. 1993).

In addition, petrographic, grain-size, mineralogical, and chemical analyses of architectural materials (mudbricks, plasters, mortars, constructed floors, etc.) has enabled identification of a range of natural and anthropogenic source materials and characterisation of different manufacturing techniques (Goren and Goldberg 1991; Karkanas 2007; Nodarou et al. 2008; Simpson et al. 2006). In particular, the study of spatial and temporal variation of floor sequences, and associated occupational debris, at the Neolithic site of Çatalhöyük has provided clues for the use of space and changes in use (Matthews et al. 1996). At the same site, micromorphology is used to infer how the deposits were formed, while phytolith, mineralogical, and residue analyses are used to analyse specific components in each thin layer of the micromorphology block. By integrating these microanalytical techniques, it was possible to infer cyclical patterns in deposits and activity types in the middens of the site (Shillito et al. 2011). Along the same lines, at a Neolithic tell site in northern Greece, Makri, the micromorphological study identified two types of floors. One type were the numerous informal floor surfaces that were prepared with recycled

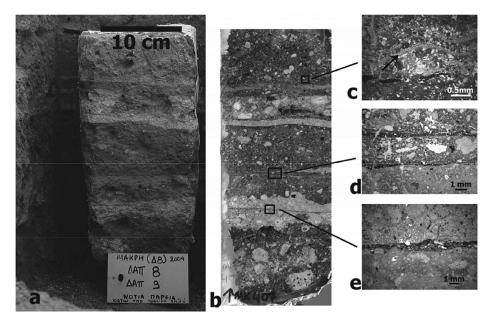


FIGURE 13.2 Micromorphological sample (a) and a resin-impregnated slab of it (b) of a series of constructed floors in the Neolithic site of Marki, Greece. Photomicrographs of certain areas of the sample are shown in c, d, and e with plane polarised light: c. Articulated phytoliths (with arrow) on top of a floor that might represent a relic of matting; d. Red clay finishing coats (dark grey on the photo) on well-prepared lime floors. The upper one was laid on a replastering; e. Well-prepared lime floors showing lamina of debris (every-day dirt) entrapped in between them (see Karkanas and Efstratiou, 2009).

rubbish by the occupants. The others were formal floors, rich in lime plaster that had been relaid at regular intervals, indicating a communal decision (Karkanas and Efstratiou 2009). Both type of floors preserve anthropogenic remains on their surface, implying specific activities (Figure 13.2). At the Iron Age Tel Dor (Israel), the microscopic fabric and content enabled differentiation between constructional fills (i.e., those representing a single depositional episode) and occupational-accumulated fills (i.e., those slowly accumulating through continuous *in situ* habitation) (Shahack-Gross et al. 2005). The nature of the fills allowed a chronological and functional association between artefacts and fills, and the surrounding architecture. Such a microanalytical approach may also solve problems related to the general nature of the site. At the Iron Age settlements of the Negev highlands (Israel), based on the nature of the components and the microstructure and arrangement of dung remains, and corroborated by mineralogical and isotopic analyses, it was shown that the inhabitants were desert-adapted pastoralists, rather than garrisoned soldiers as it was long believed (Shahack-Gross and Finkelstein 2008). Occupational deposits might have been exposed to high temperatures. Mineralogical and chemical analysis, corroborated by micromorphology, at Tel Dor identified different ways that heat-affected sediments were produced and accumulated (Berna et al. 2007). This type of analysis can be used to reconstruct fire-associated activities.

Finally, unintentionally left chemical imprints of daily activities provide important clues as to past practices and space use. Phosphorous and multi-element analyses have been applied to a range of occupational sequences with very promising results (King 2008; Middleton 2004). For example, a good correlation is found between high phosphorous concentrations in soils and food processing, consumption and disposal, whereas heavy metals are related to the use of mineral pigments and craft activities (Terry et al. 2004).

4 CONCLUDING REMARKS

Geoarchaeological research is an integral aspect of archaeological study. As Renfrew stated when he originally coined the term, "every archaeological problem starts as a problem in geoarchaeology" (1976: 2). This is not surprising if someone considers that all archaeological findings are buried in sediment or scattered on the landscape. Geoarchaeology provides the means for interpreting the context of all anthropogenic remains, either of a site or an individual artefact. In the realm of the site, geoarchaeology is of increasing importance, but a lot more has to be done to unravel the full suite of anthropogenic processes responsible for the formation of the archaeological deposits. Nevertheless, studies so far have clearly shown how powerful a tool geoarchaeology is in understanding the use of space and the nature of the human activities, or for reconstructing the palaeoenvironmental setting of a site. In studying the role of humans in changing past landscapes, geoarchaeology has made huge progress in the last few years. However, cause-and-effect relationships cannot rely only on natural science information but must also draw on social science approaches in an integrative and interdisciplinary methodology (Butzer 2005). Finally, one of the most successful applications of geoarchaeology is landscape reconstruction for finding new sites and understanding site locations, distributions and spatial changes. These kinds of studies are based on a much more solid body of geological, geomorphological and other environmental data, and - with an associated literature of far greater temporal history - have consequently more straightforward interpretations.

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Radiocarbon Dating Simon Blockley

The use of radiocarbon dating in archaeology, including sample selection calibration and quality control recommendations.

1 INTRODUCTION

The radiocarbon (¹⁴C) method underpins most chronologies for the last 50,000 years, due to the accuracy and ubiquity of the technique, suitable for organic material and some carbonates, with uncertainties in the tens to hundreds of years. It is used widely in archaeology, and in studies of past environmental change, but there have been problems to overcome. This chapter outlines the basic principles and examines some of the key developments in the technique.

2 PRINCIPLES

¹⁴C is an unstable isotope of carbon. Two stable isotopes ¹²C and ¹³C occur naturally and make up respectively ¹²C 98.89 per cent, ¹³C 1.11 per cent and 0.0000000001 per cent ¹⁴C (Aitken 1989). ¹⁴C is formed in the upper atmosphere through subatomic nuclei, themselves products of cosmic ray interaction with atoms. During production free neutrons interact with ¹⁴N and add a neutron to the nucleus in exchange for a proton:

$$^{14}\mathrm{N} + \mathrm{n} \rightarrow {}^{14}\mathrm{C} + \mathrm{p}$$

¹⁴C has a half-life of 5730 + 40 years (Aitken 1989), although originally calculated by Libby as 5568 + 30 (Arnold and Libby 1949; Libby 1955). Radiocarbon decays back to stable nitrogen in the reaction:

$$^{14}C \rightarrow ^{14}N + e^{-} + \nu_e$$

Production is controlled by cosmic ray flux, which is influenced by solar wind and the strength of the Earth's magnetic field. There is substantial variability in the cosmic ray flux and thus radiocarbon production.

3 RADIOCARBON AGES AND RESERVOIRS

¹⁴C is rapidly incorporated into the carbon cycle as ¹⁴CO₂, entering the atmosphere, and oceans (Aitken 1989). Radiocarbon enters the biosphere through plant photosynthesis and is ingested by animals through the food chain. The terrestrial biosphere is usually in equilibrium with atmospheric ¹⁴CO₂ and while living, organisms have the same radiocarbon isotope ratio as other parts of the biosphere. Upon death the organism ceases to be in equilibrium and radioactive decay reduces the radiocarbon content and therefore isotope ratio.

In marine organisms there is an added complication due to the residence time of carbon in the oceans. The deep ocean is the largest carbon reservoir and there is a long residence time. Upwelling of deep water means that old carbon is mixed with younger carbon leading to an upper mid ocean radiocarbon *reservoir effect* (R) of 400 years too old. There are regional variations in R (Δ R) of tens to hundreds of years. Due to past changes in ocean circulation there is also variation in Δ R over time. Changes in Δ R over time can be hundreds to over a thousand years (Eiriksson et al. 2004; Siani et al. 2000;). Dating marine samples, or human remains with a marine dietary component, may be difficult. Localised Δ R values have also been recorded in deep lakes or in lakes where there is input from geological carbon.

4 RADIOCARBON MEASUREMENT

In the early experiments Libby (1955) and co-workers used modified Geiger counters. This was followed by a technique where samples were either combusted or evolved to CO_2 and then analysed in gas proportional *beta* counters, with the activity being proportional to the ¹⁴C in a sample. This technique requires large samples, leaving many archaeological samples unavailable for dating. The next development involved liquid scintillation counters (Polach 1987), using liquid compounds that fluoresce during exposure to ionising radiation. Each fluorescence event is proportional to a *beta* emission. The benzene has the chemical formula C_6H_6 meaning most of the sample is carbon. A sample is initially converted to CO_2 and then into benzene, then placed in vials in a liquid scintillation counter. Activity is measured using photomultiplier spectrometers. Liquid scintillation reduces sample size and many laboratories still use scintillation counters.

For archaeology a key development was accelerator mass spectrometry (AMS) dating in the 1980s. This approach directly measures the proportion of carbon isotopes in a sample (see Aitken 1989). Samples are initially converted to CO_2 and then to graphite. In an accelerator, graphitised samples are sputtered using a suitable gun, ionised and accelerated towards a high voltage source. The ion beam enters an ion stripper, which breaks up any remaining molecules, and also strips electrons from the outer shells of the ions, reversing their charge. The now oppositely charged ions accelerate away from the source as they continue along the accelerator, where they are then separated by mass using magnets. This allows the proportion of ¹²C, ¹³C and ¹⁴C to be measured with a significant increase in instrument sensitivity and reduction in sample size. This has made radiocarbon dating suitable for a wide range of artefacts, most famously the Turin Shroud (Damon et al. 1989).

5 COUNTING ERROR

Despite the sample size differences, laboratories are capable of reporting similar measurement errors (a function of sample size, concentration, counting statistics, measurement time and instrument sensitivity). Errors are reported as a mean age with a Gaussian uncertainty one degree of freedom (1 σ ; or 68 per cent confidence). Early radiocarbon measurement errors were often large but precise estimates with errors of 10–50 years are now possible, even for older samples (e.g., Jacobi and Higham 2009).

6 FRACTIONATION CORRECTION

Natural isotopic fractionation occurs during exchange between carbon reservoirs and within organisms, which requires correction as it would produce an incorrect age. To do this most laboratories use mass spectrometers to measure the ratio of ¹²C to ¹³C in a sample, compared to a standard (δ^{13} C). Due to the abundance of these isotopes, this does not require accelerator spectrometry. This fractionation is

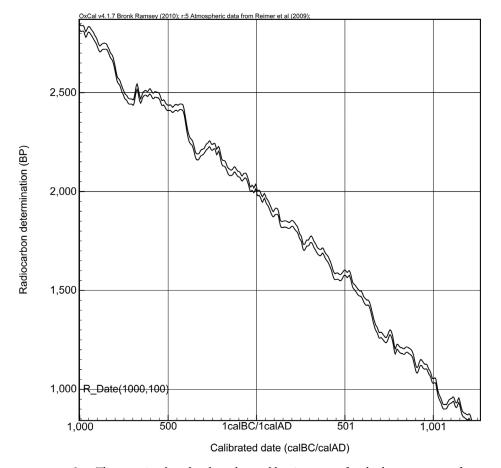


FIGURE 18.1 The tree-ring based radiocarbon calibration curve for the last 1000 years from IntCalo9 (Reimer et al. 2009) showing the fluctuations in the production of radiocarbon production over time. (Adapted from Reimer et al. 2009)

mass dependent and, as the masses for all three isotopes are known, $\delta^{13}C$ can be used to correct for ¹⁴C fractionation. The expected $\delta^{13}C$ ranges for most types of samples are also known so $\delta^{13}C$ is used to check for obvious contamination.

7 RADIOCARBON CALIBRATION

The production of radiocarbon in the atmosphere varies significantly. This means that the radiocarbon timescale is non-linear. Figure 18.1 shows the variation in radiocarbon production in the last few thousand years. Radiocarbon dates thus

require calibration against samples of known age. For part of radiocarbon time there is a well established European and North American tree-ring chronology that can be used. Tree rings vary in width due to climatic and local environmental conditions and trees can be matched statistically by their pattern of ring widths. This has been used to generate a master record to 12,400 BP (years before 1950; Reimer et al. 2009). Since the 1970s there have been attempts to use radiocarbon dated tree rings to provide radiocarbon calibration (e.g., Suess and Clarke 1976). As atmospheric CO₂ equilibrates rapidly, calibration using these data should be effective for the Northern Hemisphere at least. Moreover, as the uncertainty on tree-ring dating is very small, it is possible to produce high resolution calibration curves. Hundreds of radiocarbon measurements have been taken from the tree-ring archive with radiocarbon counting errors as low as 10-15 years. Since the 1980s attempts to produce internationally accepted curves have been undertaken, with several radiocarbon laboratories using agreed methodological protocols. The first of these provided calibration for the last 6000 years (Stuiver and Kra 1986). Successive curves have extended the tree-ring based calibration further back (Stuiver et al. 1993) and it now extends to 12,400 BP in the IntCal 09 curve (Reimer et al. 2009). In addition to the northern Hemisphere tree-ring calibration, a curve for the last 1000 years has been developed for samples that require precise dating in the Southern Hemisphere (McCormac et al. 2004).

Radiocarbon can be measured 40,000-50,000 radiocarbon years, leading to efforts to calibrate beyond the tree ring limit. In the early 1990s, IntCal93 was extended to ~19,000 Cal BP (calibrated BP; Stuiver et al. 1993) using paired ²³⁴U/²³⁰Th and ¹⁴C dates on corals. The first of these extensions, while important, was of low resolution and more coral data were included in IntCal 98. Additional resolution in the IntCal 98 and later IntCalo4 and 09 curves (Reimer et al. 2004; Reimer et al. 2009; Stuiver et al. 1998) came via radiocarbon dates on a high resolution marine core from the Cariaco basin, off the coast of Venezuela (Hughen et al. 1998). Samples of planktonic foraminifera were ¹⁴C dated and an absolute timescale was derived by comparing climate data from the core to the climate record from Greenland GISP2 ice core. The records were aligned assuming a synchronous relationship climatic record between the two sequences and the layer counted GISP2 chronology was transferred onto the Cariaco record (Hughen et al. 2004). From the end of the tree-ring limit to 26,000 Cal BP there was sufficient agreement between calibration data for a consensus calibration curve to be agreed, beyond this limit consensus broke down. This was resolved to a degree when Cariaco was realigned to climate data from $^{234}U/^{230}$ Th dated speleothems from Hulu Cave, China, which brought the Cariaco curve into line with the coral data (Hughen et al. 2007). This, along with the addition of significantly more coral data points, formed IntCalo9, allowing consensus calibration to 50,000 Cal BP.

Despite the success of IntCalo9 there were problems. Firstly, marine-based curves have a constant reservoir offset applied (Reimer et al. 2009). Attempts to compare the Cariaco radiocarbon data with other cosmogenic isotopes (10Be) from the Greenland ice cores suggested one or more shifts in ΔR at Cariaco (Muscheler et al. 2008; Reimer et al. 2009). While this problem was addressed to a degree in IntCalo9 by not using some sections of Cariaco, some researchers suggested a terrestrially-derived calibration is required. Potential data are available from either speleothems, or through long annually laminated lake records, such as Suigetsu, Japan (Nakagawa et al. 2012). These records also have their own issues, however, such as reservoir effects in carbonate rocks or years of missing annual laminations in lakes. Archaeological interpretations based on calibrated radiocarbon dates must, thus, always take a degree of caution, whether using IntCalo9 or subsequently published curves (e.g. IntCal13, IntCal19) that incorporate additional datasets. However, it is far better to attempt calibration than not. Since the development of the first calibration curves the implications for archaeology have been significant in changing ideas and allowing radiocarbon-based chronologies to be compared to other records such as ice cores and absolute radiometric techniques.

8 AGE MODELLING

Calibration adds an additional uncertainty to radiocarbon-based age models. The probability distributions of calibrated dates are affected by the shape of the curve as can be seen in Figure 18.2. It is normal to express calibrated uncertainties at 95 per cent confidence and the variability in the shape of the calibration curve means dates with the same radiocarbon error can have different final 95 per cent ranges. The non-Guassian probabilities of calibrated dates have led to significant advances in age modelling techniques. Where measurements have Guassian distributions there are a range of classical statistical techniques available. While such approaches, particularly regression, have been used to examine radiocarbon dates (e.g., Lowe et al. 1995) these are rarely statistically appropriate. One exception to this is the use of the Chi-squared test for the combination of radiocarbon dates from the same organism (Bronk Ramsey 2009). An alternative approach is the use of Bayesian

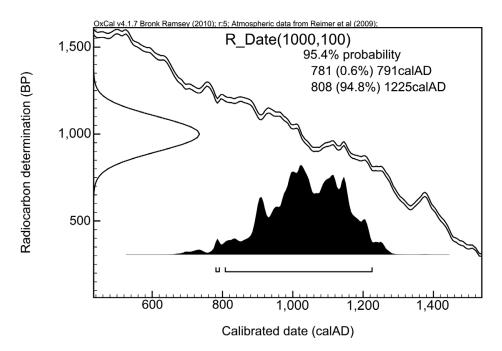


FIGURE 18.2 Calibration of a radiocarbon date and the impact of the calibration curve on the probability distribution of the final age.

analyses using complex probability matrices. This approach, pioneered in radiocarbon dating for archaeology by Buck and colleagues (Buck et al. 1991; Buck et al. 1992), has been very successful in generating useful age models and is now widely applied (e.g., Blockley and Pinhasi 2011; Buck et al. 1996) with a range of Bayesian calibration programmes freely available (e.g., OxCal, Bronk Ramsey [2008]; BCal, Buck et al. [1999]).

The Bayesian approach is based on the incorporation of prior information to constrain likelihood in a posterior highest likelihood density function. The prior consists of the radiocarbon data, the calibration curve, stratigraphy and any rules imposed, such as the uniform prior, where within set constraints any outcome has equal likelihood, see Bronk Ramsey (2008) and Buck et al. (1996). Models include dates in sequence, where the constraints are that age must not decrease with depth, and more advanced Poisson distribution models of sediment formation (Bronk Ramsey 2008). These methods have been used successfully in a range of settings including caves (Jacobi and Higham 2009) and lakes (Blockley et al. 2004; Blockley et al. 2008b). Other models include phases, which are unordered groups useful for modelling multiple phases of occupation at a site.

9 PRETREATMENT AND QUALITY ASSURANCE

Quality assurance can be broken down into the following questions: (1) are the age of the sample and the event of interest the same; (2) is there the potential for contamination during the time of burial; are there effective protocols to (3) remove and (4) avoid contamination in the laboratory?

1) Are the Age of the Sample and the Event of Interest the Same?

This question relates to the care taken during field investigations. The importance of correct sampling and the choice of suitable materials cannot be overstressed. For example, many archaeological sites are dated by charcoal samples often from hearth deposits. It is essential to know how this fits into the stratigraphy of the site and to have the charcoal identified to plant species, as wood from some trees can be hundreds of years old at the time of burning. In some areas this led to a programme of dating human, animal bone or bone artefacts. While archaeologically sound, this strategy raises further problems relevant to questions 2 and 3, as bone is a complex material with significant potential for contamination. Some environmental remains give good dating results but care has to be taken in establishing secure context and determining there is no reworking. A careful strategy based on a large data set from different types of suitable samples is often seen as the way forward (Lowe et al. 1995).

2) Is There the Potential for Contamination during the Time of Burial?

This is a significant issue in radiocarbon dating. In most archaeological contexts charcoal is relatively robust with a straightforward pretreatment process. While this does not always hold, charcoal is often the sample of choice, but wood and most macrofossils also give good results. Bone is prone to *in situ* contamination and extensive pretreatments have been developed. In environmental samples, macrofossils, such as seeds or plant material are often seen to be less prone to contamination than other options. However, movement within a profile is not uncommon for such material. On the other hand, taking samples from bulk peat, while stratigraphically more secure, is open to a much wider range of contamination including percolation of humic acids down a profile or in-wash of dissolved dead carbonate.

3) Are There Effective Protocols to Remove Contamination in the Laboratory?

Most material that is routinely dated has suitable pretreatment strategies. For many archaeological sites that date within the last 10,000 years, samples such as wood or charcoal will receive effective pretreatment. Radiocarbon laboratories have developed some important steps such as δ^{13} C. There has, however, been a long-running debate over effective pretreatment for archaeological bone, and to a lesser extent old charcoal. Effective bone dating started in the 1980s with access to accelerators, allowing laboratories to date collagen, the main protein in bone (Hedges and van Klinken 1992). Even then, despite major dating programmes in the early to mid-1990s (Housley et al. 1991; Housley et al. 1997), important samples were problematic. Recently some laboratories have specialised in producing bone ages. This involves testing for viable collagen using the carbon/nitrogen ratio of the organic component (Brock et al. 2007) and using ultrafilters to remove and discard material other than large collagen strands (Higham et al. 2006).

Charcoal is usually pretreated by heating and cleaning in acids and bases (ABA). For old samples this is not necessarily reliable (see Bird et al. 1999 and Chappell et al. 1996) and some laboratories have developed a harsher pretreatment by aggressive oxidation of samples to remove organic contamination (ABOX; Turney et al. 2001). While not yet as routinely used as ultrafiltration, such techniques have the potential to refine the chronologies of older sites.

4) Are There Effective Protocols to Avoid Contamination in the Laboratory?

Radiocarbon laboratories test reliability using known standards as well as international comparison exercises, where samples are sent to them as part of the exercise and used to test measurement reliability, error estimation, and pretreatment protocols (Scott et al. 1998). Despite this rigour, radiocarbon dating is challenging due to minimal natural ¹⁴C concentration and the difficulty of removing younger or older carbon contamination.

Quality Assurance Procedures

In order to use and understand radiocarbon dating it is important to follow good practice. As a guide, below are some of the common criteria used by internationally accepted studies.

- 1) Good security of association between sample and dated event, considering reworking and long-lived species.
- Good understanding of the depositional context, including problems such as ¹⁴C reservoirs.
- 3) Rigorous removal of contamination (Brock et al. 2007; Brock et al. 2010a; Brock et al. 2010b; Higham et al. 2006).
- 4) An internationally agreed calibration curve is available and calibration uncertainties are accounted for.
- 5) Statistical manipulation of the radiocarbon data should account for the nonnormal probability densities (e.g., Buck et al. 1991; Buck et al. 1992).

10 CASE STUDIES: KEY TRANSITIONS IN HUMAN HISTORY

A prevailing question in archaeology is how and when major transitions occur. One example is the colonisation of Europe by anatomically modern humans (AMH) and the apparent replacement of the Neanderthals (H. neanderthalensis). Despite debates over hybridisation (Duarte et al. 1999; Tattersall et al. 1999) and recent genetic evidence for limited interbreeding (Green et al. 2010), there is a clear pattern of a disappearance of the Neanderthals as a distinct species and a transition from Middle to Upper Palaeolithic tools some time around 40,000 years ago (Joris and Street 2008) or later. This pattern is based on a large database of radiocarbon ages, of varying quality. An added interest is the role of climate change, as around this time cold episodes known as Heinrich (H) events are recognised in a variety of archives, including Greenland (Svensson et al. 2008). One of these episodes, H4, dates to around 40,000 BP, and is suggested by some as being significant in the AMH replacement of Neanderthals (Fedele et al. 2008). It has also been suggested by d'Errico and Sanchez-Goni (2003) that H4 delayed AMH arrival into southern Iberia, as the reduced biomass of arid southern Iberia may have limited their ability to occupy this region, creating a Neanderthal refuge.

If the available radiocarbon dates are taken and simply calibrated, using IntCalo9, then there is significant overlap in many regions between late Neanderthals and early AMH. Indeed in some regions the raw data suggest many thousands of years of overlap. This has contributed to the idea of some cultural and possibly genetic admixture. Evidence for this idea comes from transitional tool industries, such as the French Châtelperronian, which has both Upper Palaeolithic and Midddle Palaeolithic affinities, but is thought to be a Neanderthal industry. Gravina et al. (2005) and later Mellars et al. (2007) suggested intrastratification of Châtelperronian and Aurignacian levels, suggesting sequential periods of use by Neanderthal and AMH groups. Others, however, see Neanderthal extinction as very rapid (Fedele et al. 2008) and linked directly to the H4 event and possibly to a very large volcanic eruption known as the Campanian Ignimbrite (CI) from the Campanian volcanic province, Italy, at 39,282 \pm 110 BP (de Vivo et al. 2001).

There is a problem of radiocarbon chronological control over this important research area. An example of the problem is the age of the iconic human remains of the Red Lady of Paviland. This male Palaeolithic burial comes from the Gower peninsula, Wales. Initial dating in the 1980s using early AMS facilities suggested that the burial occurred around 18,000 radiocarbon years ago. Recent re-dating using more advanced pretreatment put the date back to $26,350 \pm 550$ BP (Aldhouse-Green and Pettitt 1998). Further re-dating using the ultrafiltration technique, has moved the burial back to between $28,870 \pm 180$ to $29,490 \pm 210$ (Jacobi and Higham 2008). In this case the type of pretreatment has made 10,000 years difference in the reported age, and is much more acceptable on archaeological grounds. If we consider that the radiocarbon database for the Middle/Upper Palaeolithic contains hundreds of dates on bone (Joris and Street 2008), with few using ultrafiltration, then the difficulties of studying this period are apparent.

In addition to bone, many of the available dates for the Middle to Upper Palaeolithic transition are also based on charcoal, and old charcoal dates may be problematic. As mentioned above, at this time there is a major volcanic eruption know as the CI, that is very well dated by non-radiocarbon methods. Ash from this eruption is widespread and is found in archaeological sites as far afield as Italy and Russia (Pyle et al. 2006). Ash deposition can be treated under normal circumstances as a geologically instantaneous event and thus be used to link the chronologies of numerous sites. Blockley et al. (2008a) used this fact to test the available radiocarbon chronologies for Palaeolithic sites where this ash occurred. They combined the IntCal 09 calibration data, and the available radiocarbon dates for the sites containing the CI ash, the ⁴⁰Ar/³⁹Ar ages for the CI from the volcano and a Bayesian model to order the stratigraphy of the sites. This study revealed that in all of the Middle to Upper Palaeolithic archaeological sites where the ash was reported all but one of the radiocarbon dates were too young to support the model. This suggested that, firstly, these sites required re-dating using ABOX pretreatments, and that all charcoal ages for this time period had to be suspect. Subsequently Higham et al. (2009) re-dated samples from this time period at the important site of Grotta di Fumane, Italy, using the refined acid-base-oxidation/stepped combustion (ABOX-SC) method for

charcoal pretreatment and demonstrated that this method systematically produced older and more reliable ages than the traditional ABA pretreatment.

What is clear is that one of the most important transitions in European history is currently poorly understood and that this is the result of problems with the available radiocarbon database. New work is showing that significant improvements can be made but it is now important for the relevant archaeological community to drive research forward by a European wide re-dating programme.

Another major transition in European and Near Eastern history is the onset of agriculture (e.g., Childe 1951), starting in the Fertile Crescent and the southern Levant. This has been seen as a three-stage process with the adoption of a fully sedentary lifestyle by Natufian hunter-gatherers; followed by a process of innovation in the Pre-Pottery-Neolithic PPNA-C (Bar-Yosef 1998; Kuijt and Goring-Morris 2002). This occurs roughly between ~17,000 BP and ~9000 BP, and coincides with climatic upheavals at the end of the last ice age. There is a long period of cool, and in the Levant dry, conditions, followed by a sudden warming at the start of the late glacial period (~14,700 BP), with a rapid return to cold conditions during the Younger Dryas (12,800-11,700), before warmer stable conditions return into the Holocene (11,700 BP; Rasmussen et al. 2006). It has been argued that these conditions induced fluctuations between cooler/dryer and warmer/wetter conditions in the southern Levant, influencing human populations. In this model, late glacial Natufian hunter-gatherers adopted a fully sedentary lifestyle, but are forced adopt a more open mobile lifestyle by the Younger Dryas (the Late Natufian), and that this may have been the earliest onset of horticulture, before a transition to early agriculture in the PPNA (Bar-Yosef 2001; Bar-Yosef and Belfer-Cohen 2002).

From an environmental perspective this is sound as there is good evidence from speleothem records in Israel of a pattern similar to the record from the Greenland ice cores (Bar-Matthews et al. 1999; Bar-Matthews et al. 2000). However, all of the important sites in this period have chronologies based on radiocarbon dating. In order to investigate the chronological aspects in more detail, Blockley and Pinhasi (2011) took the existing radiocarbon database for the southern Levant (Pinhasi et al. 2005), and examined it on quality assurance grounds. One of the most interesting results was that, from a database of 149 samples, only 84 were deemed suitable (based on internationally accepted criteria) and 9 of the most important sites had no reliable dates covering this transition. Importantly, rejection was not based on any prior idea of what a correct age should be but simply the application of basic quality assurance approaches.

11 CONCLUSION

This chapter has aimed to outline the key principles, advantages and challenges of radiocarbon dating. This method is at the heart of archaeological chronologies and thus deserves the careful attention of archaeologists and archaeological scientists. When archaeological interpretation is undertaken without sufficient attention to chronological problems, it is very easy for poor interpretations, or at least confusion, to be the result. The method is powerful and applicable to a range of sites and samples, but care and attention is required from the sample selection stage through to the final interpretation of dates, or of large radiocarbon databases, to ensure accurate and meaningful dating of archaeological events.

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