KEYWORDS: ***Mud-wasp nest*** *–* ***Rock painting*** *–* ***Micro-charcoal*** *–* ***Radiocarbon*** *–* ***OSL dating***

DATING MUD-WASP NESTS ASSOCIATED WITH ROCK ART

Alan L. Watchman

Abstract. Dating mud-dauber wasp nests associated with paintings in rockshelters is examined. The nature and sources of materials of wasp-nests and the radiocarbon and luminescence methods and assumptions used in the dating processes are reviewed. The relationship between the ages of individual micro-charcoal particles in the muddy sediments and the nest-building period is considered. Also stressed is the absence of direct links between micro-charcoal in a wasp-nest, the period of building nests and the application of paint to a rock panel. Dating micro-charcoal from a nest generates an average value for the age of all carbon particles in the sample, and the range in age is unknown. The lapses in time between nest construction, degradation, cementation, and paint application are unknown and cannot be determined.

Introduction

Typically, the time when an artist applied charcoal or paint to a rock surface is determined by measuring the radiocarbon content of the paint medium, such as plant fibre binders (Cole and Watchman 1992; Watchman and Cole 1992), animal fats (Brook et al. 2018), mucilaginous juices (Watchman et al. 2002; Gutiérrez 2013) and oxalate in paints (Russ et al. 2017; Steelman et al. 2021). Beeswax resin has been dated reliably (Nelson et al. 1995; Watchman and Jones 2002). The dating of re-precipitated calcite, amorphous silica and oxalate mineral layers containing carbon and uranium-bearing components under and over a layer of paint is another way of estimating maximum and minimum ages (Arsenault et al. 1995; Aubert et al. 2014; Watchman 1993, 2004; Mazel and Watchman 2003; Steelman et al. 2021). However, there is an issue in the dating of charcoal. For example, charcoal on the floor of the Chauvet Cave, Ardèche, France, is about 30 000 years old (Quiles et al. 2016), so today, someone can make a drawing dated to that age.

The research by Gillespie (1997) is fundamental to understanding the potential problems of dating rock art using direct or indirect means. He re-evaluated the dating of what was initially regarded as blood associated with rock art painting at Laurie Creek, Northern Territory (Loy et al. 1990; Nelson 1991, 1993). Gillespie (1997: 436) concluded, 'if direct radiocarbon dating of the abundant rock art-work in Australia is to be done, other organic residues must be found which: (a) can be unequivocally tied to the event of painting the rock, (b) contain carbon contemporary with that event, (c) can be extracted, purified, identified, and (d) are present in sufficient quantity for this to be reliably done'.

One such alternative carbon-bearing residue is the remnant of mud-wasp nests. Mud-dauber wasps often build their nests in rockshelters and some nests may be directly associated with rock paintings. However, the temptation to determine the *terminus post quem* and a *terminus ante quem* (see Langley and Taçon 2010) for a rock painting by dating the carbon-bearing contents of mud-wasp nests ignores serious scientific concerns. Recently, Finch et al. (2021) published dating results using the carbon in mud-wasp nests to estimate the ages of some Australian rock paintings, but the conclusions of their study are controversial. The reasons why their results are debatable stem from the protocols, presumptions and assumptions of the approach adopted in their earlier work (Finch et al. 2019).

Finch et al. (2019) problematically assume a direct relationship between the age of the carbon in a mud-wasp nest and the age of an associated painting in a rockshelter in the Kimberley region of Australia. They conclude from their analyses and radiocarbon dating of carbon in mud-wasp nests that 'The wide range of ages measured establishes that, at the millennial scale, the wasp nests have been built quasi-continuously in the Kimberley over at least the last 20 000 years and are, therefore, capable of providing age estimates for archaeological features and rock art throughout that period' (Finch et al. 2019: 153). The conclusion is incorrect because of a propositional fallacy. Their own results provide evidence countering their assumption.

Finch et al. (2019: 151) state that 'some of the samples, however, were single nests where all carbon should be of much the same age and yet the age differences were still significant'. The authors seem surprised all carbon particles are not the same age in a single nest, and they add to their disbelief and self-deception by also declaring 'even with the revised pretreatment protocol there are significant age differences between the heavy and light fractions in 15 of the 16 heavy/light sample pairs analysed'. The authors ignore the facts. They attribute the divergences to 'intermittent flooding acted to thoroughly bind younger detrital material to the mineral matrix so that it tended to sink during HLS (heavy liquid separation), moving more of the younger carbon into the heavy fraction' (Finch et al. 2019: 152). The authors also concede, 'the age differential between fractions is plausible, even if the reason why the four light fractions are older is not certain' (Finch et al. 2019: 152). The authors admit they do not understand why different carbon fractions are not the same age, yet they dismiss the contradictory results and errors to assert the validity of their approach to the dating of rock paintings using micro-charcoal in mud-wasp nests.

Bednarik (1979) recognised and acknowledged that the carbon system of all rock sub-surfaces is open. Consequently, he (Bednarik 2014) cautioned against targeting a painting event by neatly bracketing it between the minimum and maximum ages derived from direct dating endeavours using radiocarbon. Indeed, they bracketed it, but the intervals between them may be so great that the result is of limited practical use. There is no scientific reason to suggest the average age of the micro-charcoal in any particular mud-wasp nest, as asserted by Finch et al. (2019, 2021), approximates the age of a painting underneath because there is no chronological relationship between the ages of micro-charcoal particles in a nest, the nest-building activity, and the application of paint by an artist to the rock surface. The reasons for this statement are outlined below.

The sources of mud for building nests

The mud used in the construction of the nests is gathered by female mud-dauber wasps from damp silty deposits and carried in their mandibles to rockshelter panels. During the repetitive process, the wasps do not necessarily collect mud from precisely the same location. Little detailed information is available about the range of the flights taken by the wasps for building nests. The simplest approach assumes the muddy sources are close to the rockshelter and less than a kilometre (Naumann 1983; Camillo 2002). The clay and silt that cements the mud nest can be found in river valleys. Some of the sediment is of local origin, but some has been carried downstream and is augmented by carbonaceous materials of various ages that originated upstream. Thus, muds are inevitably composed of components of a wide variety of ages. Even at the same site, mud may be derived from the erosion of unconsolidated sediments in a moist alluvial terrace and also from recent flows and deposits on the stream bed contiguous with the eroding terrace slope.

The rockshelters in Australia's Kimberley, Arnhem Land and Cape York Peninsula regions have formed in hard silicified sandstone, quartzite and limestone (Needham et al. 1973; Wende, 1997; Brocx and Semeniuk 2011). In the tropical north of Australia, mud sources include stream deposits along and across river channels, black soil plains, margins of billabongs, flood deposits and the eroding banks and foot slopes of incised streams. These sites contrast with the frequently coarse sandy soils in the rugged sandstone and quartzite terrains near rockshelters. Though Finch et al. (2019: 151) noted, 'wasps were observed collecting mud from five sites; all within rock shelters. Two sites were on the sides of small ephemeral pools of water. Of the other three sites, one was a muddy slope in a very dark cavity deep within the rock shelter.' However, Naumann (1983) describes the usual sources of suitable mud as the abundant clay and silt alluvial deposits found along moist stream banks and riverbeds. Floods add to the complication by eroding ancient riverbanks upstream and depositing fine-grained older sediment components derived from elsewhere. Micro-charcoal contained in muddy sediments may therefore not have been deposited concurrently with the enclosing silt. Thus, there is significant uncertainty in the temporal relationship between the age of the micro-charcoal in the mud and the nest-building event.

The composition of the mud-wasp nests varies

The mud used for building nests is not only composed of cementing clay and silt, but also fine-grained sand and a wide array of trace ingredients: pollen, spores, sponge spicules, phytoliths, micro-carbon particles, natural sugars, starches, various organic compounds, and parts of grasses and other plants (Bednarik 2014). After the young wasps have hatched and broken through the outer walls of their cells, the remaining parts of nests gradually degrade, leaving stumps or stubs of the original nest. Often some of these fall off completely, but some harden and remain attached to the rock panel. Various rock surface chemical processes achieve cementation of the residual nest components. Amorphous silica from seepage water may be deposited on rock faces before and after a nest is built. The water containing silicic acid emanates from slow seepages out of joint and bedding planes and between quartz grains. As it flows across rock faces and evaporates, amorphous silica precipitates and encapsulates insect sclerites, charcoal particles and fragments of vegetation (Watchman 1992). Diatoms may also live where a constant flow exists. These carbon-bearing components can be used to determine the approximate radiocarbon ages of thin laminations of amorphous silica skins associated with rock paintings (Watchman 2004).

Whewellite and weddellite oxalate minerals (Del Monte et al. 1987) may crystallise from the reaction between oxalic acid and various hydrated salts that are precipitated on the surface by evaporation of moisture (Hernanz et al. 2007; Sturm et al. 2015). The sheltered, damp and dusty rockshelters are ideal habitats for fungi and bacteria, some of which excrete oxalic acid from their metabolic processes (Watchman 1991; Russ et al. 1996; Di Bonaventura et al. 1999; Burford et al. 2006). The combination of micro-organic and evaporitic processes in northern Australian rockshelters leads to the crystallisation of hydrated sulphate, nitrate, phosphate and oxalate minerals (Hughes and Watchman 1983; Green et al. 2017). These salts accumulate in thin films and encrustations and may cover the stubs of wasp nests. The carbon-bearing oxalate salts may contaminate the dating of micro-charcoal, but this can be readily overcome by using appropriate pretreatment chemical processes. X-ray diffraction, infrared spectroscopy, scanning electron microscopy and other geochemical analyses may help resolve issues regarding the identities, sources and ages of the micro-charcoal particles selected for age determination. More importantly, they assist in decisions concerning the chemical treatments necessary to minimise contamination of the carbon.

The nature of the painting

Rock art researchers are understandably keen to determine when paintings were made. However, the formidable challenge is to determine the precise timing of the application of each paint layer to a rock panel. Potential carbon-bearing components in pre-Historic paints include saliva and urea (unreported), plant juices (Arocena et al. 2008), blood (Loy et al. 1990), charcoal (Pepe et al. 1991) and plant fibres (Cole and Watchman 1992). However, most of the painters chose finely pulverised haematite (Fe_2O_3) and more rarely jarosite ($KFe_3(SO_4)_2(OH)_6$) for the reddish paint and did not use an organic binder (Chalmin et al. 2003; Huntley et al. 2013), but combined them with water as the medium. If present in sufficient quantity and if they can be decontaminated, then the carbon compounds can be dated using the radiocarbon method.

Although some critics may say that red laminations in encrusted rock surface coatings result from the natural staining caused by iron-rich water, the application of reddish iron-rich pigment and a medium onto bare rock is an anthropogenic action. The artist may or may not add an organic binder to make paint. The red pigment itself does not contain any usable carbon for radiocarbon dating unless the paint medium is organic rather than water, so indirect means are routinely used to bracket the paint layer between two precisely measured intervals (Watchman 1993; Aubert et al. 2014). An equivalent scenario exists where oil or acrylic paints are applied to canvas or wood and then coated by varnish (Townsend and Keune 2006). In effect, a paint sandwich is produced, and in a geological sense, the paint forms a mineralised deposit on a micro-unconformity. The lapse in time between the covering of a rock surface by the paint and the natural processes taking place on the rock panel, including the construction of wasp nests, is incalculable.

Using the age of micro-charcoal in mud-wasp nests to date the associated rock paintings presumes the duration of the micro-unconformity is short, but the precise length of elapsed time is unknown. A layer of paint over a mud-wasp nest may erode and give the impression that the wasp built her nest over the painting. However, without making a cross-section through the rock, mud-wasp nest and paint layer, it is impossible to determine with confidence whether the paint is situated under or over the nest (but see Finch et al. 2019: 146, Fig 4b). A similar micro-unconformity exists where a nest is built over a paint layer. Again, the time lapse between the application of the paint and nest-building is immeasurable. Such studies of cross-sections are typically used in art history in the forensic analysis of oil paintings on canvas and wooden boards and for the study of frescoes. Using microscopes and cross-sections makes it possible to determine the micro-stratigraphic relationships between multiple layers of paint and varnish or rock substrate, mud-wasp nest and paint. Without such detailed analyses of cross-sections of all dated samples, the physical relationships between the mud-wasp nest and a layer of paint can only be estimated.

Sampling of mud-wasp nests and paint layers

From the earliest research into the dating of rock paintings (Watchman 1985), the procedure adopted for removing part of a painting for analysis has been to select a partly detached flake of painted rock. This method minimises damage to a painting because of collecting only the portion of the fragile surface deemed likely to fall off naturally. However, the most appropriate samples are generally not obtained for study. Removing a partly detached flake requires close scrutiny of the rock surface and then delicate levering a loose rock fragment onto carbon-free aluminium foil. Whereas taking a stub of a mud-wasp nest for analysis and dating involves chipping the hardened residual mud from the rock panel. The method described by Finch et al. (2019: 141) is 'Most mud wasp nests were removed using a 6 mm chisel, sharply tapped with a small hammer and caught in a sheet of aluminium foil'.

Bracketing a painting in time by indirect dating of mud-wasp nests, therefore, requires the removal of many small stubs, and this action across a panel of paintings might be regarded as vandalism. Physical removal of the stub and paint may therefore be considered irresponsible and unethical, even with the permission of traditional owners and cultural heritage authorities. Furthermore, the nest material collected is destroyed during the radiocarbon dating process, so the opportunity for applying innovative dating methods on those samples is forfeited.

Radiocarbon dating of micro-charcoal in mud-wasp nests

Finch et al. (2019: 140) point out, 'radiocarbon dates on different organic components (e.g. wood, charcoal, pollen, plant matter) within a sediment sample have been shown to differ significantly because they originate from multiple sources of different age.' Not only are the various components of different ages, but the micro-charcoal particles do not come from a single source. The radiocarbon age of micro-charcoal in a nest indicates the average time of death of the trees, grasses and other plants from which the micro-charcoal originated. The measured age has nothing to do with transporting material by the wasp from a muddy site to the rockshelter. There could be no relationship at all.

Rock art investigators should not assume that the age of the mud, based on the average age of multiple micro-charcoal particles and the nest building event, are coeval or approximately contemporaneous (Finch et al. 2019). This is because mud may have been sourced at various times from various locations and sedimentary deposits containing carbon of multiple ages.

There is an additional problem. A radiocarbon age is not measured from a single grain of micro-charcoal but from many grains in a nest, all of marginally different ages. The calculated age estimate of micro-charcoal from a nest is, therefore, a pooled mean average of the ages of all the tiny carbon-bearing particles in the sample. Charred organic matter is found in soils and sediments and charcoal (highly resistant to decomposition due to its condensed aromatic composition), is routinely used by investigators (Cohen-Ofri et al. 2006; Eckmeier et al. 2009) as a marker for past natural and anthropogenic fire events (Patterson et al. 1987). In soils and sediments, micro-charcoal is derived from grass fires and the breakdown of macro-charcoal (Magid et al. 1996; Stevenson 1994: 496; Sollins et al. 1996). Micro-charcoal consists of stable, resistant carbon components and can be dated with ^{14}C accelerator mass spectrometry. However, the heterogeneous nature of charcoal and the possibility of surface oxidation and degradation from larger fragments means that multiple micro-charcoal samples from a distinct sedimentary layer may consist of numerous chemical compositions. Those different compounds will likely yield disparate ^{14}C ages. Exacerbating the issue, the age of a piece of charcoal does not date a fire event but the assimilation of radiocarbon by a particular part of a living tree (Bednarik 2000: Fig. 1). The time delay or inbuilt age between the life of a plant and charring could be centuries (Schiffer 1986) or, with the involvement of micro-organic activity, even millennia.

Thus, micro-charcoal particles in sediments are influenced by external factors over millennia. The unknown processes and events affecting them will impinge on their age distribution within a mud sample or another sediment. The micro-charcoal fraction might contain more stable, recalcitrant aromatic carbon compounds and therefore yield older ^{14}C ages, but that may not necessarily be the case. To determine a reliable age of a rock painting associated with mud-wasp nests, the coeval relationship between the micro-charcoal in the mud and the period of constructing nests must be demonstrated, not simply presumed.

That problem may seem easily addressed by dating several 'modern' mud-wasp nests and measuring the radiocarbon age of the micro-charcoal within them. Micro-charcoal in modern nests will not be contemporary with current or recent nest-building but reflect past processes. There may be a difference of one or two millennia or more depending on many unknown factors. Researchers should not presume carbon particles in mud that had been incorporated into mud-wasp nests are derived from contemporary events when, in fact, such materials may have originated from sources variously laid down during environmental changes over several millennia.

Heavy liquid separation of micro-charcoal with different densities yields heavy (sinkers) and light (floaters) fractions. The sinkers are particles of recalcitrant resistant carbon compounds that have persisted in the environment despite biogeochemical processes. Floaters, also resistant to degradation, comprise different carbon structures than their sinker counterparts (Plaza et al. 2019). The process of partitioning carbon into two fractions, then determining their radiocarbon ages adds another level of complexity to the problem of dating rock paintings. Particles in both fractions will range in age unless the carbon components of the sediment are in a closed system. Given the non-uniform environmental factors, the heavy and light micro-charcoal fractions will likely have disparate ages. This is because different geomorphological and biogeochemical processes will have affected the sediment (Lehmann and Kleber 2015).

Heavy and light fractions with similar age determinations indicate the silt used for building nests did not come from sediment of mixed components. Contrarily, disparate ages for the two fractions signal various components (Hassink and Dalenberg 1996). The resulting radiocarbon average ages for the two fractions in a mud-wasp nest, nevertheless, have no temporal connection with the construction period of mud-wasp nests. They only indicate the range in ages of the micro-charcoal in the light and heavy fractions of the sediment. The results may bear no relationship to the age of an associated painting.

The real question falls back to the female wasps. Where did they obtain the mud, what was the carbon age in the different mud pellets, and when did they build their respective nests? In summary, dating micro-charcoal from a nest generates an average value for the age of all carbon particles in the sample, and the range in age is unknown. Moreover, to reinforce the facts, the lapses in time between nest construction, degradation, cementation, and paint application are also unknown and cannot be determined.

Luminescence dating

Optically stimulated luminescence (OSL) refers to the release of energy by crystalline solids when exposed to light (Kennedy and Knopf 1960; Aitken 1994; Murray and Wintle 2006). Ionising radiation from naturally occurring minerals containing potassium, uranium and thorium release electrons in the crystallographic structures of quartz and feldspar grains. Electrons become trapped in crystal lattice defects associated with impurities or chemical substitutions. The build-up in charge over time (the palaeodose) depends on the nuclear radiation flux to which the grains were exposed. The excess energy of these trapped metastable electrons is released by green/blue light (approximately 500 nanometres) and measured as photons. The amount of energy released is a function of the time since the mineral grains were last exposed to light (bleached).

In practice, the antiquity of an ancient mud-wasp nest is estimated from an OSL analysis of an aliquot of quartz grains of the hardened stub of a nest (David et al. 1997; Roberts et al. 1997). The γ-ray dose is derived mostly from the local bedrock, the cosmic-ray dose rate is estimated at each site, and the β-particle dose rates from the nests are deduced from X-ray fluorescence and α-particle spectrometry (Roberts et al. 1997). The elapse of time since bleaching during nest-building can be calculated from the dose rate and measuring the amount of accumulated excess electron energy.

This article further argues that the fundamental problem with using OSL to date the building of mud-wasp nests is the presumed solar bleaching of all the quartz grains during transportation by a wasp. Those innermost quartz grains in a wad of mud collected by a wasp may not fully reset the OSL clock but retain a residual dose (Bednarik 2014). If all the grains are unbleached of their electron energy (even using single-grain analysis), then the measurement of luminescence age will skew the estimate to an older value.

Other uncertainties arise. Considerable luminescence bleaching of the quartz grains may occur after a mud-wasp nest has deteriorated (Sanderson et al. 2011; Sohbati et al. 2012) because of the penetration of sunlight through the corroded stub. Also, the geochemical processes acting in the near-surface environment may lead to the separation of radioactive parent and daughter nuclides in the components of a nest. This disequilibrium influences the estimate of the radiation dose affecting the quartz grains. Also, radon is produced from the decay of uranium in the mud, and rock mass adjacent to the sample and its radioactive energy will impinge on quartz grains. Similarly, the estimation of cosmic radiation is difficult because of the shielding effect of rock shelter geometry. The consequences of these luminescence processes will distort the assumed constant radiation rate of the quartz grains and misrepresent the age of nest construction.

Conclusion

Using mud-wasp nests to determine the age of associated rock art depends on knowing the composition of the mud and the lapse in time between the painting event and nest building. The direct physical relationship between the nest and the layer of paint also must be demonstrated. The radiocarbon age of micro-charcoal in a mud-wasp nest represents an average of the ages of all the carbon-bearing particles in the mud. It indicates the range in age of the sedimentary components, not the nest-building event. Carbon particles in a nest may not be contemporary with nest building and will therefore be unrelated to the painting of a rock surface. If the stub of a mud-wasp nest contained the remains of a spider or a dead larva, then a more reliable age could be obtained for nest construction. Such a scenario is possible but unlikely because of the destructive nature of the taphonomic processes. Therefore, using micro-charcoal in mud-wasp nests is unreliable for determining the age of an associated rock painting.

Hand-picking grains or using heavy liquids to separate charcoal particles from a nest before radiocarbon dating aids in eliminating possible carbon-bearing contamination. It does not solve the problem of determining when a nest was built. Dating two heavy liquid-separated fractions of different ages confirms the diverse nature of mud collected by the wasp. The result confirms the heterogeneous composition of the mud. Two charcoal fractions of comparable ages substantiate the homogeneous nature of the mud but say nothing about when the nest was built or the age of an associated painting.

Using a combination of materials and methods involving mud-wasp nests does not provide a reliable age estimate for a painting under a nest. An investigator may consider using oxalate, micro-charcoal and OSL to determine the age of the nest components. However, the OSL approach assumes the quartz grains were completely bleached of luminescent signals before incorporation in the nest and not altered subsequently. The radiocarbon approach assumes all the micro-charcoal in a nest is only marginally older than the nest-building period. Therefore, using mud-wasp nests for the dating of rock art depends on many assumptions. The validity of those assumptions is easy to ignore but difficult to test. Dating micro-charcoal and quartz grains in mud-wasp nests using the radiocarbon and luminescent approaches leads to inaccurate and unreliable results. The indisputable fact is that no temporal relationship exists between the time of flight of a female mud-dauber wasp and the age of the components in the mud of her nest. Publication by Finch et al. (2019, 2021) raises an alarm concerning the scientific standards of the respective journals and of their review processes, as well as the questionable decisions by the ARC panel (Australian Research Council Linkage Projects LP130100501 and LP170100155) to allocate resources for the misleading and meaningless radiocarbon numbers for charcoal in mud-wasp nests that are unrelated to rock art.

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Dr Alan L. Watchman
College of Humanities, Arts and Social Sciences
Flinders University
Social Science South
GPO Box 2100, Adelaide 5001, S.A.
Australia
alan.watchman@flinders.edu.au

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