Title: Reflectance: current state of research and future directions for archaeological charcoal: results from a pilot study on Irish Bronze Age cremation charcoals

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Highlights

- This study is a first attempt to use the reflectance method to measure absolute burn temperatures from charcoal of archaeological cremation burials.
- Reflectance as a method for estimating charcoal burn temperature has been under investigation for some time. To date studies have used modern pre-charred wood samples (formed under a variety of conditions) to establish basic performance parameters and calibration curves to apply to charcoals recovered from both archaeological and modern contexts.
- The method shows promise in its application to archaeologically recovered charred materials, especially wood, although to date, only a small number of studies have been completed.
- Reflectance results of the charcoal did not demonstrate the range of expected temperatures associated with cremation (ca. 650°C to as much as 1000°C). A variety of explanations are considered.

- In particular, proof of this method's utility in archaeology will require better rationalisation of the calibration curves used to date as these currently represent a variability of typically 100-150°C (and up to as high as 180°C) for any one reflectance value
- Un-sieved soil samples should be collected routinely for this method to gather the smallest charcoal fractions

Keywords

Reflectance, charcoal absolute burn temperature, reflectance calibration curve, cremation

Suggested reviewers

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The maximum difference of 1.81 (at 500 °C) represents a potential variation of up to 180 °C (i.e. +/- 90 °C). This is the most extreme variation, and most fall within a range representing a variation of ca. 100 °C (i.e. +/- 50 °C).

Abstract

'Reflectance' is a method that estimates the absolute burn temperature of charcoal from the 'shininess' of resin mounted samples. The method's usefulness for archaeological charcoal is yet to be comprehensively studied. This report details first results from reflectance testing of archaeological charcoals excavated from Irish Bronze Age

cremations, which included calcined bone. As calcination of bone commences at 650 °C, it was expected that the charcoals would reflect at least this temperature. This was not the case for taxonomically identified charcoals >2mm, nor for micro-charcoals of c. 250µm, although measured temperatures rose slightly with decreasing fraction size of charcoal remains. Depositional practice, combustion completeness and taphonomic influences may have all played a part in this result, and these will need careful consideration in different archaeological circumstances. However, the greatest challenge for reflectance of archaeological materials lies in obtaining full agreement on the production and use of reflectance calibration curves. Current calibration curves differ substantially, by 100-150 °C (+/- 50-75°C) *and in one instance up to as much as 180* °C (+/- 90°C). *Without better agreement on calibration, the method's ultimate usefulness in archaeological research will be limited. At the level of refinement currently possible, it will still be useful for determining very high or very low temperature processes, and possibly the difference between charcoal fuel and raw wood fuel fires. The latter has distinct implications for estimating ancient forest wood consumption, since more wood is consumed in processes employing charcoal fuel. Proving the utility of reflectance for archaeological purposes may also require modification of normal practice for archaeological field collection of charcoal, to include collection and laboratory processing of un-sieved soil samples.*

1 Introduction

In the last forty years, the systematic study of archaeological charcoal has greatly increased our knowledge of past environments as well as socio-economic activity relating to fuel collection and consumption (Asouti and Austin, 2005; Chabal, 1992; Chabal *et al.*, 1999, Dufraisse, 2006, Théry-Parisot *et al.*, 2010, 143). In the laboratory, charcoal analysis has been substantially limited to taxonomic identification, and more recently, estimation of cropping indicators by examination of annual tree ring patterns, see for example: Ludemann (2006), Veal (2012), Marguerie and Hunot (2007) . More recently, attempts to further characterise modern charred materials in terms of their chemical and physical characteristics have been made in experimental procedures designed to assist archaeologists in their interpretation of ancient charred remains (Braadbaart *et al.,* 2009; Braadbaart *et al*., 2012; Braadbaart *et al.,* 2016; Chrzavzez *et al*., 2011; Chrzavzez *et al*., 2014, Lancelotti *et al*., 2010).

Reflectance testing has been attested as a useful tool for demonstrating approximate burn temperatures of modern, and some archaeological charcoal (Braadbaart and Poole, 2008;

McParland *et al*. 2009a,b; McParland *et al.,* 2010; Scott and Glasspool, 2005). A more detailed explanation is provided in the supplementary material. (LINK HERE to Supplementary material 1) McParland *et al.* (2010) have in particular, demonstrated the almost linear relationship of reflectance of specially prepared samples of charcoal across a range of wood species, temperatures, and charring times (as opposed to earlier work which concentrated on one or two species, and sometimes limited charring times). Extensive testing of the reflectance method over a range of archaeological depositional types however has not yet been carried out. In this study, charcoals of a range of size fractions from cremation burials were evaluated, since the expected temperature for successful human cremation is inferred to reach *ca.* 650°C as a minimum (if calcined bone is observed (Wahl, 1982:27.)). One domestic pit fill and two hearth contexts were also evaluated as a comparison.

2 Background

2.1 Reflectance of modern charcoals

Wood charcoal is formed through the heating of wood in the absence of oxygen, and can be formed intentionally (in the manufacture of charcoal fuel), or as a by-product of wood-fire burning. Soil examined from archaeological sites normally contains a mixture of charcoal (incomplete burning of raw wood, or 're-burning' of charcoal fuel) as well as ash (the remains of wood burned to completion in the presence of oxygen). Morphological, physical and chemical properties of charcoal can differ depending on two groups of variables associated with the heating process. The first group consists of heat related variables, which include temperature, time of exposure and heating rate (°C/min). The second group consists of wood property variables, which include taxon, size, thermal conductivity and other variables that can change during the charcoalification process itself, see for example: Braadbaart *et al.* (2007), Braadbaart and Poole (2008), and Asouti (2007).

The connection between increasing temperature formation and increasing mean random reflectance value (studied in polished blocks under oil) is relatively well established (Braadbart and Poole, 2008; McParland *et al*., 2007; Scott and Glasspool, 2005). The reflectance (%Ro) of charcoalified organic material provides information regarding the absolute temperature to which the material in question has been exposed. This reflectance is quantified and measured by comparison with the reflectance of known standards, achieved through experimental work. The most comprehensive work on reflectance of modern woods (McParland *et al*., 2009a) measured temperatures of modern wood samples burned under a variety of controlled circumstances. Mean reflectance measurements carried out on different species (*Quercus, Corylus, Acer, Fraxinus, Betula, Pinus, Erica, Calluna* and *Ulex*) corresponded closely to the original temperatures at which the modern charcoal was produced. This choice of woods is particularly pertinent to ancient fuel studies as these taxa are very commonly observed in archaeological assemblages. This method discriminated changes in reflectance levels at intervals of 50 °C ranging from 300 °C to 1100 °C. It proved (as did earlier studies) that a near linear relationship exists between charcoal burn temperature, and average reflectance measurements, regardless of time of heat exposure and at a variety of temperatures (McParland *et al.,* 2009a, 2010) (figure 1). Reviewing published calibration curves shows that while the linear trend is always demonstrated, for any individual reflectance measurement, agreement among the curves as to the associated temperature can vary by as much as 100-180 °C (Section 5.1).

Fig 1 Calibration graph of reflectance vs burn temperature developed from charring of modern oak wood under a variety of temperatures and times (modified from McParland 2010)

Experimental work on modern charcoals produced under laboratory conditions, provides the backdrop for the present study, with the reflectance curve for *Quercus* developed by McParland (2010) (also discussed in McParland *et al.* (2009a)), acting as the calibration curve. If reflectance is to be of use in an archaeological setting, then we must exclude the possibility that taphonomic processes undergone by archaeological charcoal will dull or obliterate the reflectance signal of the charcoal remains and/or recognise when such limitations may be present. If this concern can be allayed, then the technique may help establish actual burn temperatures under different technological conditions (for example, metal smelting, ceramics and glass production), and thus improvements in technology (as represented by higher heat processes). It should also be possible to discriminate between charcoal and raw fuel fires.

2.2 Irish Bronze Age Cremation

During the Bronze Age in Ireland, cremation was the predominant rite of treatment for human remains (Lynch and O'Donnell, 2007, 105). The most likely form of the structure may have been similar to that more visibly attested in later historic periods, for example on coins from the Roman period (Toynbee, 1971, 32), and those pyres used even today on the Ganges in the Hindu rite of cremation. Pyres are typically built by alternating and increasingly smaller levels of logs built in a roughly pyramidic shape. The cremation process would have been challenging in prehistory, depending as it does on time, temperature and oxygen (Mc Kinley, 2000, 404).

The maximum temperature achievable in the combustion process will be affected by the size, shape and quality (i.e. calorific potential) of the fuel; the structure of the pyre, (or hearth or kiln); the body weight and fat content, ambient weather conditions, and the supply of oxygen. A rough (but imperfect) proxy for calorific potential of a wood is its specific gravity (a measure of relative density at a particular moisture content (Veal 2012, 33-34). In general, denser woods such as *Quercus* and *Fraxinus* produce a longer-lasting source of heat than less dense woods such as *Salix, Populus* and *Alnus* (Gale 2003, 36). In the study area *Quercus* appears to have been selected for cremation pyres during the Bronze Age, possibly because of its high calorific potential (O'Donnell 2011; 2016).

Heat is not a fixed value for any one wood type since it will vary with moisture conditions, size and shape of logs burnt, and other ambient factors in combustion (Lyons *et al.*, 1985). We speak of calorific potential in any fire process, as the actual calorific return achieved will depend on the amount of heat value transferred to the object of combustion. In open pyre cremation, a large amount of heat is lost to the atmosphere. After cremation has completed, Irish research has shown that a sub-sample of bone, charcoal or a mixture of the two was taken from the funeral pyre and buried, within urns in grave pits, or directly into grave pits (Lynch and O'Donnell, 2007). Charcoal from this study is derived both from pyres and from cremation graves. Taphonomic processes differ between the two contexts, for example in the pyres, wood would have burnt *in situ*, suggesting that samples could be taken from the centre and outskirts of the pyre. This may result in differing reflecance values, charcoal at the periphery of the pyre may have burnt at lower temperatures than the centre. In any single cremation event, however, bone and wood from a cremation pyre may have origins from anywhere in the fire, from the centre, to the periphery and have been exposed to varying fire temperatures. As burning progresses, fuel, both burnt and unburnt, can potentially move around in the fire due to a range of agents, for example, differing temperature patches in the fire will be present due to the varying flammability of materials present; oils or perfumes thrown onto the fire will momnentarily increase temperature, and once temperature reaches body fat ignition point, burning will progress more quickly. (LINK TO Supplementary material 2). Fatter body parts (and their nearby fuel) may be more likley to burn and drop through the pyre first. Observation of remains from some urns suggests a range of sizes of charcoal are present at deposition. Upon excavation from urns or pits, the origins of the recovered charcoal (in terms of its position in the cremation pyre) cannot be determined, nor may its time spent in the pyre be estimated (McKinley 2008, 167-68). While not the case at this study site, sometimes bones are found arranged vertically in anatomic order within urns, however even in these (rare) cases, it is unlikely the charcoal is also so organized. This would be exteremely difficult to carry out, but also, due to charcoal's lightness and fragility, settlement will continue as the urn is being filled, carried and potentially even after deposition. McKinley (2008) also notes that in her experiments, roughly 700-900 kgs of wood are required and the main process of combustion occurs in about 2-3 hours, with the pyre being left overnight to cool. Even after 8 hours, some of the body may still remain unburnt.

2.3 Archaeological background

Templenoe, Co. Tipperary, Ireland is the largest Bronze Age flat cemetery excavated in Ireland (figure 2) where the remains of 89 grave pits, 57 of which contained cremated bone were excavated. Charcoal results from six graves and one potential pyre are presented here (figure 3). Four possible pyre ventilation pits were also identified in association with the burials. *In situ* burning was not evident, but they are classified as potential pyres due to their location in the site and their larger dimensions than the grave pits. Other domestic features, potentially unrelated to the cemetery include pits and postholes, indicative of settlement activity. The cemetery was in use from the Early to the Middle Bronze Age (dated by AMS radiocarbon determinations) (Mc Quade *et al* 2009, 130-133).

Fig 2 Map of location

Fig 3 Cremation pits and possible pyre pits, looking southwest. (Doody 2008, p. 117)

Charcoal was analysed mainly from the grave and potential pyre pits, of which seven samples are included in the present study. Seven native Irish wood species were identified, dominated by *Quercus* (deciduous only, there being no evergreen *Quercus* in Ireland), and Maloideae. Nomenclature follows Stace (1997) . The other taxa present were *Fraxinus, Corylus avellana, Ulmus, Prunus avium/padus* and *Alnus*.

Human skeletal remains were identified from 31 pit features from Templenoe. The majority of the bone deposits within the cemetery contained less than 10g of cremated bone, with an overall range of between only 0.08g and 697g, suggesting that token deposits of bone only were buried, based on the average weight of a cremated adult individual (Mc Kinley 1993). Four non-adults and 20 adults were present while one male and one female were identified (Geber 2009, 213-215). Modern studies have shown that temperatures ranging from 650 to ≥ 800°C are required to successfully cremate human bone (i.e. until the bones are whitish to white in colour) (Wahl 1982, 27). In archaeological samples however, the reliance upon colour as an indicator of exposure temperature essentially an imprecise criterion both because of individual differences in the ability to perceive fine colour distinctions and because burnt bones may change colour if they are buried (Shipman et al 1984). A variety of colours are often observable in the remaining bones, and a complex interaction of many factors can influence colour, and thus it cannot always be a reliable indicator of temperature (Devlin and Herrmann 2008). Some scholars have even noted that cremation may have occurred in a lower temperature range in pre-history, from about 500-600 °C (Barber 1990; van Andringa et al. 2013, Vol 1:8-9). Ignition starts from about 350 °C, with sufficient oxygen and flammable material. In the process of cremation, temperatures of up to 1000°C can be reached. Starter materials may have included brushwood, oils, perfumes, and of course, textiles. These would all burn to completion and thus may be lost to the archaeological record.

The people at Templenoe were good at cremating their dead. Taking various caveats into account (Devlin and Hermann 2008), the grey-white colour of the majority of the collected bone fragments in the samples indicates successful cremation. The bones exhibit the fourth and fifth category of degree of burning, according to Wahl (1982, 21), and Geber (2008), suggesting burning temperatures of 650-800 ºC. This corresponds most closely with burn colour codes 5 and 6 as described by Steiner and Kuhn (1995). Therefore, the samples should provide a useful control with which to compare the burning temperatures as measured through reflectance. As a further comparison, reflectance values were also measured from charcoal from one domestic pit at Templenoe and from charcoal from two nearby Bronze Age domestic hearths, at Lissava and Ballylegan (figure 2) (Mc Quade *et al* 2009).

3 METHODS

3.1 Processing and identification of charcoal

Soil samples were processed by flotation (O'Donnell 2007, 28). Charcoal was identified following known standards (Marguerie and Hunot 2007; Schweingruber, 1978) and a modern reference collection.

3.2 Subsampling of charcoal from available material

Small (*c.* 2cm longitudinally) charcoal fragments, as well as fine charcoal dust were sub-sampled and reflectance measurements taken as follows:

- Charcoal was examined to measure absolute burn temperatures achieved in cremation pyres with successful (Reflectance Sample (RS) numbers 1, 3, 4, 5, 7,9, 19, 22 and 24), and less successful cremations, (RS 2, 6, 25, 26 and 27), as denoted by bone colour.
- Charcoal from domestic fires was tested as a comparison with the cremation pyre charcoal to examine differences in temperature ranges (RS 8, 11, 12).

Some differences are notable within the sub-soils from the three sites, although these are not thought to have affected charcoal preservation. At Templenoe, the sub-soil was dark boulder clay (Doody, 2008). At Lissava, the sub-soil was an orange-yellow gravely, sandy clays (Molloy, 2007). At Ballylegan, the sub-soil was compact, yellow brown sandy clay (Mc Quade, 2007). Soil pH was not recorded at the sites. Modern experimentation has shown that highly alkaline environments (such as may exist from high concentrations of ash from combustion of wood) can weaken charcoal structure, suggesting lower reflectance values may be observed (Braadbaart *et al.,* 2009). Ash not noted from Templenoe, and the context descriptions of the cremation deposits are very cohesive, indicating they were filled with loose, black silty clay (Doody, 2008). Even if high alkalinity were present at burial it is difficult to know the rate/range of pH changes that may have occurred due to percolation of rainwater, and/or groundwater over time.

3.3 Reflectance testing in two stages

Taxonomically identified charcoals >2mm identified were roughly crushed and mounted in one of two methods (cold set, or hot set epoxy), highly polished, and inspected at x1,000 magnification using a reflecting Nikon microphot microscope. Fifty measurements were taken and averaged for each individual sample. The hot and cold set epoxy methods have different utility depending on sample size and other factors, but a control test revealed results were not affected by setting method. The samples here were prepared both by cold set (>2mm samples), and hot set (250μm-2mm samples). In the hot set method, careful selection of the ratio of charcoal dust to epoxy powder is needed. Too much epoxy powder results in insufficient charcoal at the sample interface; too little epoxy results in a gritty sample that is difficult to polish. A ratio of about 1/3 charcoal dust to 2/3 epoxy mix was found to be suitable for these samples. (Further method details can be found in Supplementary materials 1)

Testing of samples of individual and mixed taxa was carried out. In a second round of measurements, smaller charcoals of various fraction sizes were tested (from the same contexts). Table 1 details the characteristics of the charcoals tested and the reflectance results. Sample numbers are not contiguous as some failed to be ‘readable’ for reflectance after preparation due to the challenges of sample preparation, in particular, the need to provide at the end of the process good exposure of the charcoal to the reflecting laser, i.e. at the very uppermost surface of the resin.

3.4 Reporting reflectance results: average and maximum temperatures

Standard procedure in past reflectance testing has reported ranges of average temperatures (calculated from 50-100 measurements of each individual sample). Here we follow convention, but also consider more critically, the maximum temperatures observed for each sample. Temperatures were inferred using a 1, 6, 12 and 24 hour calibration curve developed by MacParland for *Quercus* (figure 1). Results are expressed as a range of temperature e.g. a sample with a reflectance value of 5%Ro would need to be charred at 880 ^{o}C for one hour or 800 ^{o}C for 24 hours giving a range of 800-880 ^{o}C. The *Quercus* (deciduous) curve, was adopted as this was the most common wood observed within the Irish archaeological samples. No experimentation has been carried out on evergreen oak (a factor of relevance for Mediterranean data) and a wood that is usually harder and of higher specific gravity.

Table 1: Context types, characteristics, and reflectance results of tested charcoals (using McParland reflectance curve in fig. 1)

4 RESULTS

4.1 *Summary*

In the first round of readings from >2mm sized charcoals, the average temperature varied from a low of 360-410°C (Samples 7 and 10) to a high of 390-450°C (Sample 12) (table 1). The highest temperature observed was 525°C (Sample 3). Little or no variation could be correlated with wood species. As these readings were well below those expected (650-800°C), a decision was made to seek smaller fraction charcoals (by way of dry sieving the extant archaeological material). McParland *et al.* (2009a and b), Braadbart and Poole (2008) and others suggest that due to the increasingly brittle nature of charcoals with increased charring temperature, higher reflecting charcoals may be limited to the smallest size fraction. Careful sieving was made of the sub 2mm charcoals into 2-1mm, 1mm-500 µm, and 500-250 µm fractions (however, subsequently these sub-divisions provided no extra information). As the flotation material kept was processed over a 250 µm mesh, it was possible to examine material as small as this, but no smaller, from the same contexts from which the identified charcoals arose. It cannot be proven that the charcoals in the range 250 µm - 2mm are the same wood types, but it is highly probable (in any event, wood type was determined to have little bearing on the process). It should also be borne in mind that the size of a particular charcoal fragment may not only be due to fire process, but also to depositional and taphonomic phenomena, and excavation and post-excavation handling. All result in further fragmentation of archaeological charcoal.

In the second round of readings from the >250 µm – 2mm fractions, average temperatures ranged from a low of 360-410°C (Sample 26, correlating with Samples 2 and 27) to an average temperature high of 390-450°C (Sample 19, correlating with Sample 9). The highest temperature recorded was 515°C (Sample 27, correlating with Samples 2 and 26).

Thus the lowest and highest average temperatures are the same from the >2mm, and >250µm – 2mm fractions (360-410°C/390-450°C). In some cases, the >250µm – 2mm fraction actually provided a lower average temperature than the >2mm fraction (for example Sample 26, correlating with Sample 2). A slight temperature increase can be noted however, within the average temperatures of the smaller fraction samples than the larger ones as shown in table 1. Observation showed that the difference between 'successful' and 'less successful' cremations as determined by bone colour could not be explained by a difference in the calorific potential of the woods.

One of the questions considered was whether changes in temperature would be noted from different archaeological contexts. Would a cremation pyre of mixed wood taxa (a specialised construction with increased body fats) burn at a higher temperature than domestic fires of mixed wood taxa? To test this, controls were taken measuring reflectance values from Templenoe (Grave pit F179, Domestic pit F33), Lissava (Trough pit F12) and and Ballylegan (Hearth F446). Average reflectance results from the mixed taxa group of the grave pit from Templenoe (Sample 5) were 370-415°C, and these do not differ to those obtained from the domestic hearth at Ballylegan (Sample 11). Results from the domestic pit at Templenoe F33 (Sample 8) are slightly higher, at 375-425°C. The highest temperature from this comparative group is 390-450°C (Sample 12) from the trough fill at Lissava.

Table 2 summarises the maximum temperatures observed for each deposit type, comparing contexts containing bone, with those that did not, or were the controls of hearth and trough. Inspecting this table shows that generally the contexts with bone have slightly higher temperatures, the maximum reading for contexts with bone was 525°C, and for those of the controls/ no bone: 490°C, but the difference is marginal. More data, and/or employing a reflectance reading strategy that uses Rmax proper (i.e. with the transverse section aligned perpendicularly for the 'best' possible reading), as opposed to %Ro (average random readings, on unaligned sections) may provide higher temperature readings.

Table 2: Comparison of maximum temperatures observed in contexts with bone, and without bone

5 DISCUSSION

5.1 *Reconsidering calibration curves*

The temperatures measured using reflectance on the cremation deposits are lower than expected. This may be linked to the current available calibration curves.

A number of studies have published calibration graphs of the temperature / random reflectance relationship with a positive correlation between increasing temperature of formation and increasing mean random reflectance (%Ro) (Ascough *et al*., 2010; Braadbaart and Poole, 2008; Bustin and Guo, 1999; Guo and Bustin, 1998; Jones *et al.*, .1991; McParland *et al.* 2007; Scott and Jones 1991; Scott and Glasspool 2005).

The curves are in broad agreement, but they were each constructed using different woods and under a variety of conditions. Woods studied included: (conifers) *Sequoia sempervirens*, *Pseudotsuga menziesii*, *Picea abies* and *Pinus sylvestris;* (decidous broadleaves); *Quercus robur, Fagus sylvatica, Corylus avellana* and *Alnus glutinosa.*

The two curves developed by Bustin and Guo (1999) and Guo and Bustin (1998) aimed to measure Rmax (i.e. by carefully orienting the samples to provide a transverse surface exactly perpendicular to the incident light), while the rest of the studies examine %Ro (average random reflectance – without special orientation of the sample).

McParland (2010, table 10.1) provides a detailed table comparing these studies, however, among all of theses studies, it is only her own that attempts measurements across five taxa, while all the others create curves using just one taxon. Comparison of the studies is further complicated by the fact that different researchers formalise calibration measurement points at different temperatures. Table 3 summarises the maximum variation observed in %Ro where four or more comparanda readings are available, except for those measurements from 700-1100°C, where the low reading is always from McParland (2010), while the higher reading is from Braadbart and Poole (2008). Figure 4 illustrates the comparison of these latter two curves, and exemplifies how at one %Ro, quite a range of temperatures are theoretically possible. These are the only two calibration curves which to date examine oak. Differences in the reported mean random reflectance at a given formation temperature and duration may be accounted for by variable factors in different experiment designs, such as length of seasoning time of the calibration wood, dimensions of the sample material charred, or differences in charring protocol (for example, charring under nitrogen atmosphere, which results in complete exclusion of oxygen; or charring by wrapping in aluminium foil, a 'low' oxygen method). It may be that differences in polishing level, and/or calibration of differing laboratory reflectance systems may also account for these variations, as well as the fact that all measure %Ro (i.e. the average of the maxima and minima observed – without perfect alignment of the transverse plane). In figure 4, Braadbart and Poole's 2008 curve, the reflectance values as observed in this experiment result in temperature readings that can be as much as 150° C higher for the same reflectance value. McParland *et al.*'s (2010) preparatory methods however more closely mimic actual ancient fire conditions: low oxygen charring, over a variety of time periods, and, importantly, across a range of taxa most often found in European charcoal assemblages, thus this curve has been used in calibrating the reflectance results for this study. Calibration curves built on, for example, sequoia, or other conifers (summarised well in Scott *et al.* 2014:section 2.4) may have little relevance to European conditions (although sequoia of course may be useful in American studies). A further new curve is found in Hudspith (2015:3) who also measures reflectance of multiple woods types**:** *Betula nana***,** *Picea mariana*, *Picea glauca***,** *Betula papyrifera* and *Populus tremuloides* (figure 5)**.** These taxa were chosen for their relevance in the modern wildfire study to which she applies her curve~~.~~ The resinous nature of conifers (as opposed to hardwoods) may affect calibration due to the possibile recondensation of resins during charcoalification, however no recognisable pattern of reflectance calibration curves could be discerned that distinguished conifers from broadleafed species, although wood specific gravity did seem to have some influence (higher SGs give higher reflectance at a given temperature).

Table 3: Variation in reflectance measurements from six different studies (from McParland 2010, table 10.1). Studies of: Ascough *et al*., 2010; Braadbaart and Poole, 2008; Bustin and Guo, 1999; Guo and Bustin, 1998; Jones *et al.*, .1991; McParland *et al.* 2007; Scott and Jones 1991; Scott and Glasspool 2005. Table shows %Ro where four or more comparanda readings were available, except for those measurements from 700-

1100°C, where the low reading is always from McParland (2010), while the higher reading is from Braadbart and Poole (2008).

As noted, Braadbart and Poole (2008) is the only other study which derives a curve from *Quercus,* but they used very small cubes of wood which were charred only for an hour (conditions unlikely to mimic the long process of cremation), however, their remarks on this curve bear some examination. Firstly, they note that a higher rate of increase of reflectance (and therefore temperature) lies in the range 600-850° C (so their curve is not completely 'flat'). They also note a tailing off of reflectance values after 850° C, and at this temperature we may expect (as they note) that if full combustion has occurred, then in all likelihood there will be very little carbonised material to observe in the imperfect and variable conditions of reality (as opposed to laboratory) burning. Testing of small microcharcoals from inside iron slag (Veal *et al.*, in preparation), has had some success however, with reflectance values translating into temperatures over 1000°C. The fact that the 'best' calibration curve may not in fact be linear, is also demonstrated by the most recently published work by Hudspith *et al.* (2015, Fig. 5) whose fitted curve is S shaped., but whose raw data points closely follow the patterns of McParland (2010) and Braadbaart & Poole (2008)

Figure 4 : Braadbart and Poole's (2008) calibration curve and that of McParland (2010). Both derived from *Quercus,* although with very different preparation strategies.

Figure 5 : Hudspith *et al.*'s (2015) calibration curve for five experimentally charred (American) boreal woods. Mean random reflectance under oil (Romean) and standard deviations represent all species. Derived from charring small pieces of wood for 1 hour each at varying temperatures.

According to the calibration curve we believe to be most appropriate to the burning conditions of cremation (Mc Parland , 2010), the results (average temperature high of

390-450°C) show that charcoals collected above 250 μm from known pyre contexts have not demonstrated the temperatures associated with cremation (above 650°C as previously discussed). A range of variable results were obtained, with, in some cases a small upward trend for the smaller fraction charcoals. Reading from alternatives curves, such as that of Braadbaart and Poole (2008), however, the temperatures expected may be inferred in a range around and above 650° C. This demonstrates how the calibration curve used can greatly influence results and calls for greater agreement in calibration curves.

A number of explanations may be considered:

- Is the reflectance method capable of measuring temperatures in archaeological charcoals as high as 650-800°C? Experimental work creating charcoal under laboratory conditions indicates that temperatures up to 1,100°C can be measured through reflectance (Mc Parland *et al.,* 2009a, 253). When applied to archaeological materials, however, average measured temperature values reported to date are 330-410ºC (Mc Parland *et al.,* 2009b, 182) and 375-530ºC (Mc Parland *et al.,* 2009a, 6). Further work must be conducted on measuring reflectance values from archaeological charcoal to demonstrate the maximum temperatures the method can record.
- The collected remains may reflect fuel waste located at the periphery of the fire, (i.e. the cooler part of it), and/or which had been thrown on late in the cremation (and thus not exposed to the high central pyre temperature.) The cremated remains, ash and charcoal are however, culturally sorted before deposition in an urn or grave, which ultimately contains selected bones and some charcoal, a process which will have likely mixed the remains, so selective collection by the archaeologist of lower temperature (only) exposed charcoals seems improbable.
- Higher temperature charcoals tend to be more brittle and vulnerable to breakage, as noted, and therefore there is likely to be a bias towards lower temperature materials in any identified charcoal assemblage (the smaller the material size, the more probable it will be lost in surrounding soils, and/or not collected).
- The collected remains may have reached the temperature of cremation, but the taphonomic processes over *c.* 3500 years (such as percolation or mineralisation), have altered the reflectance of the charcoal. Fresh breaks are revealed before reflectance

measurements are taken making this possibility less probable in the case of external mineralisation, however, highly acid or alkaline waterlogged sites (not indicated here), may be problematic. Generally speaking, archaeological charcoals absorb chemicals in soils around them, and in particular water, diagenesis of charcoal does occur over long time periods, and more testing of this phenomena through chemical and physical proxies, as well as reflectance, may assist.

- While the smaller, (i.e. 250μm) charcoals mostly did reveal a slight increase, the change in average temperature was not statistically significant. Higher temperatures may be measureable from even smaller material, therefore this needs collection by way of un-sieved ash/charcoal/soil samples, although if combustion is virtually complete, no remains may be archaeologically detectable, as also noted by Braadbaart and Poole (2008) and others. This will depend on the origins of the charcoal, and the manner of collection

6 CONCLUSIONS AND FURTHER WORK

The results suggest that reflectance will need to be applied in a selective manner and results interpreted carefully in terms of cultural practice, combustion processes and taphonomy. Reflectance results outlined here (according to our chosen calibration curve) do not match those expected from cremation deposits, however, they reach the minimum temperature expected using the curve of Braadbaart and Poole (2008). We have not used this curve because its development involved preparation methods less close to those of prehistoric cremation, as already noted.

The calibration curve used here, and others published elsewhere need to be further refined so we can obtain well-tested curves for major archaeological fuel woods (predominantly hardwoods). The calibration line of best fit may well be a curve, rather than a straight line relationship. The apparent steeper rise in reflectance from about 550-600 °C , needs to be investigated, and we suggest may relate to a particular state of rearrangement of the carbon atoms and aromatic compounds within the charcoal.

Exchange of samples for testing in different laboratories will assist, together with checking of standards used. To this end it is suggested that a group of researchers be formed to test and agree a way forward.

Ultimately the science of reflectance may require nuanced calibration of curves for different taxonomic groups, and perhaps according to anatomical structure to see if resolution of estimated temperature may be improved beyond the 100-150°C range currently observed. Testing of other charred materials (such as seeds) is underway as olive and grape pressings are also found as fuels, see for example, Braadbaart *et al.* (2016), Rowan (forthcoming), Coubray *et al.* (forthcoming). The diagenesis of charcoal increases over time, and this needs further examination, perhaps by way of examining other chemical and physical propeorties of differently aged archaeological charcoal.

Combustion, once temperatures reach the high levels expected, may be wholly destructive under some fire regimes, and it may not be possible to collect any remnant charcoals that have been exposed to the high temperatures, although testing of reflectance on charcoals from an iron smelting site in Medieval Angkor (South East Asia) has shown more encouraging results, with some results ca. 1000°C (Veal *et al* in preparation). Testing of small materials would be desirable. Normal field collection of charcoal is by dry sieving over 4 or 5mm mesh, and/or flotation over 250μm or larger mesh. The absolute size of charcoal fragments for taxonomic identification purposes is usually >2mm. It will be useful in the future to collect un-sieved soil samples of ash and micro-charcoals (where present), and separate the charcoal by gravimetric or other method for reflectance testing to further resolve this issue. Small bulk samples collected by hand (we suggest approximately 500g would be adequate. These should be stored in a cool place, and not subjected to changing ranges of temperature or moisture after excavation. As this strategy also accords with that required for a range of archaeometric studies, its burden to the archaeologist would be minimal.

The method does have utility even at this level of refinement as it appears to differentiate high and low level temperature processes. Further work using the reflectance method in other archaeological context types is also required, for instance, the testing of ‘charcoal only’ fires, vs ‘raw wood’ fires, should provide contrasting temperature profiles.

ACKNOWLEDGEMENTS

The authors acknowledge the support of the CSIRO, Sydney; the Department of Archaeology, and the Australian Centre for Microscopy and Microanalysis, University of Sydney (especially materials preparation specialist, Mr Adam Sikorski); and the McDonald Institute for Archaeological Research, University of Cambridge. Excavations were carried out by Margaret Gowen & Co. Ltd, funded by the National Roads Authority, Ireland. This research was partly funded by the Government of Ireland, Irish Research Council (Project id GOIPD/2013/387) supported by the School of Archaeology, University College Dublin. Charcoal results are stored in the WODAN database at www.wodan.ie.

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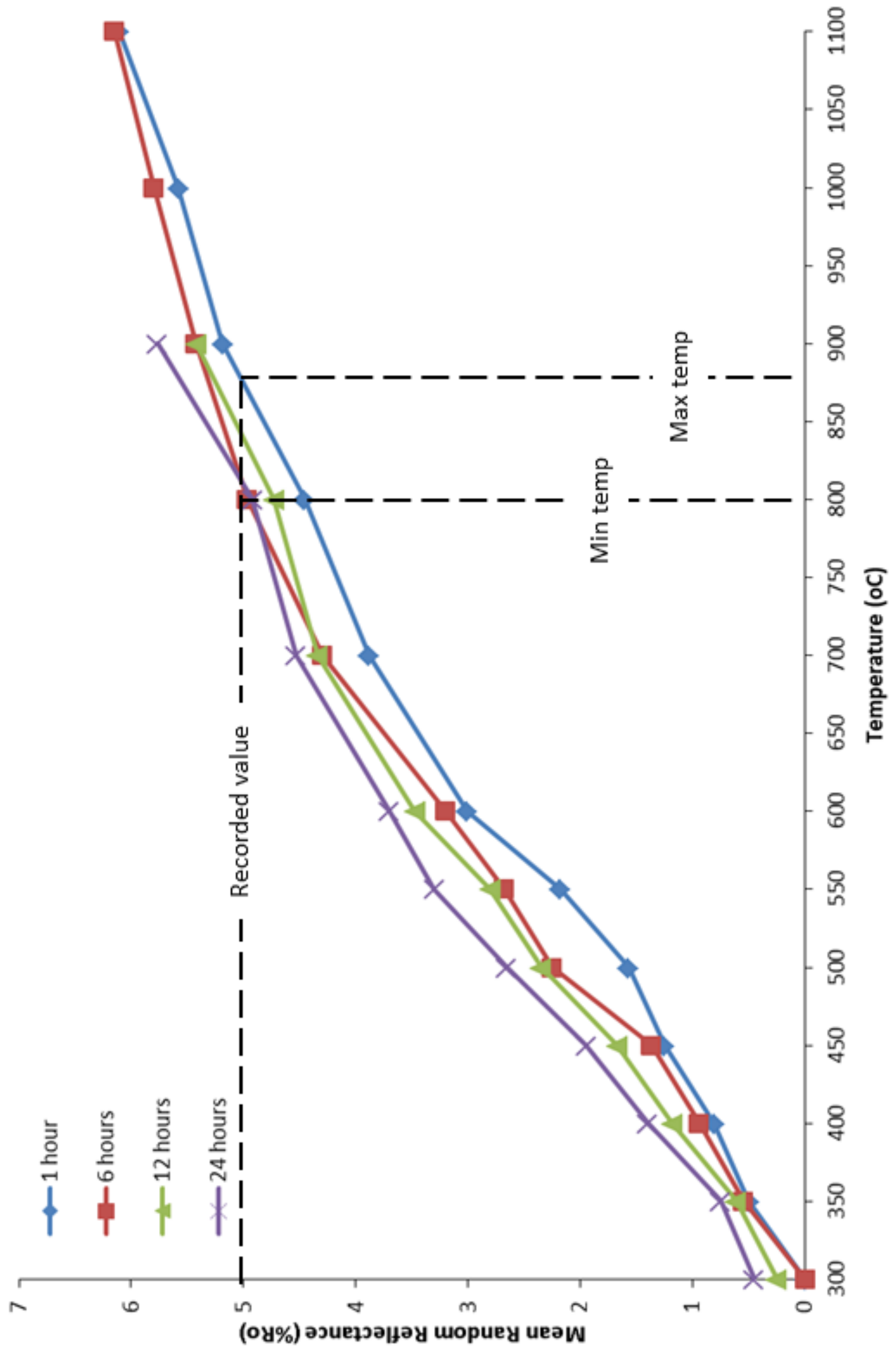
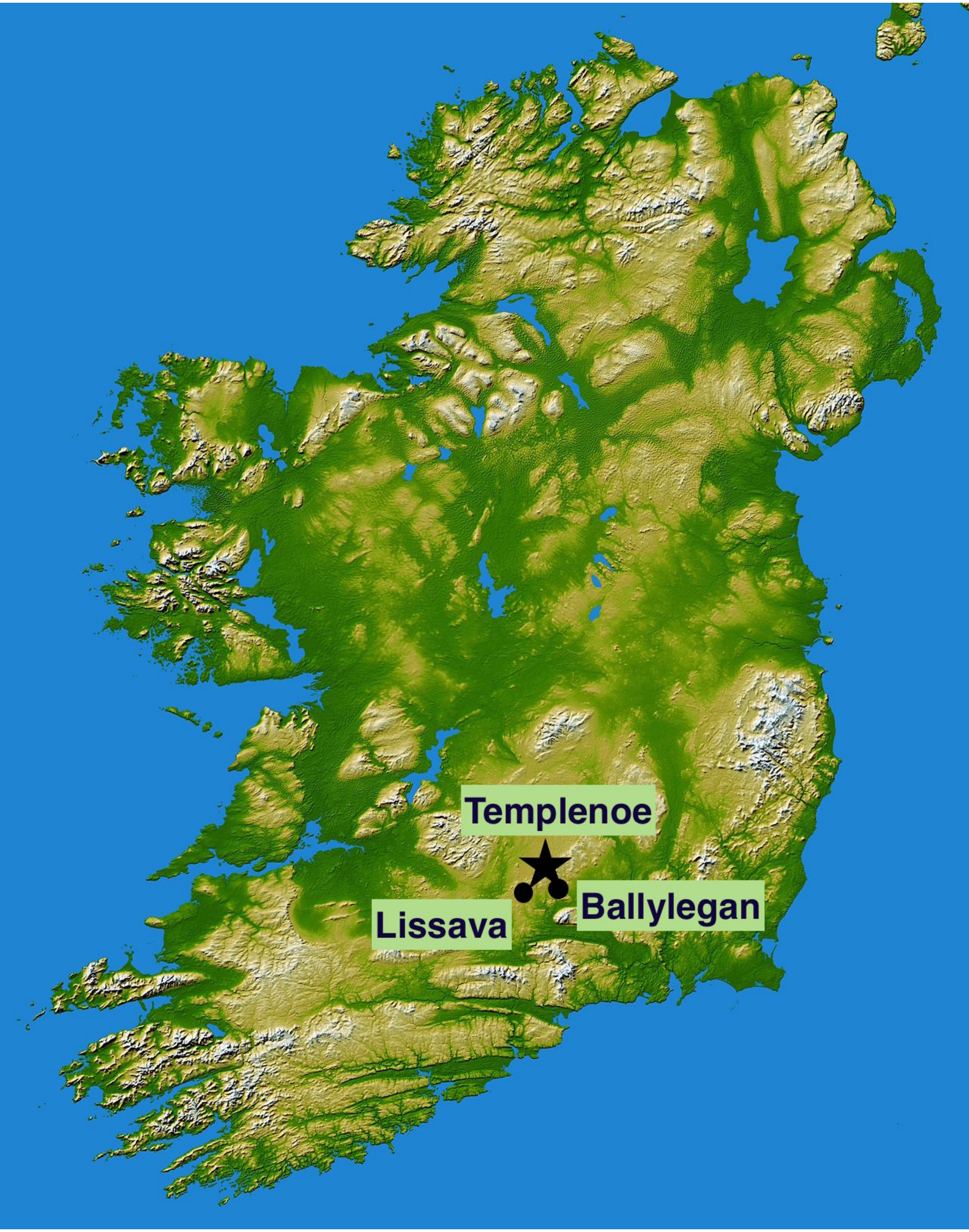
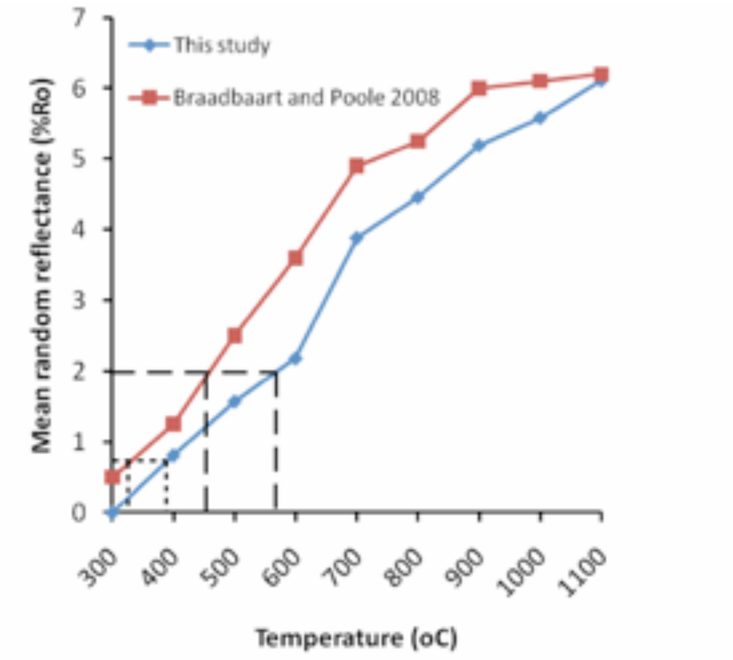
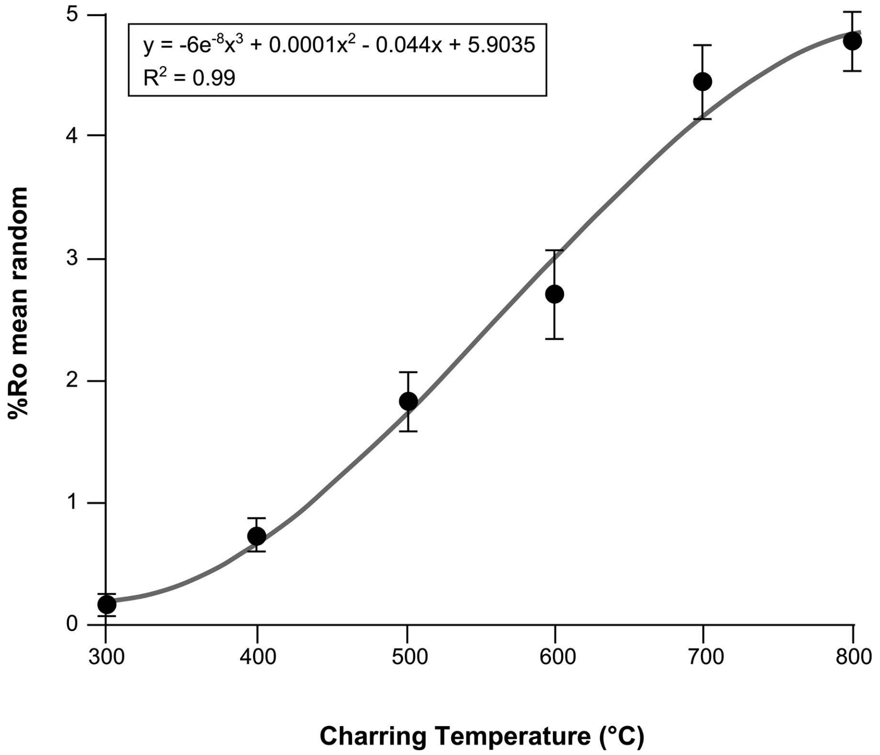

1 hour
6 hours
12 hours
24 hours
Recorded value
Min temp
Max temp
Mean Random Reflectance (%Ro)
0
1
2
3
4
5
6
7
Temperature (oC)
300
350
400
450
500
550
600
650
700
750
800
850
900
950
1000
1050
1100


Templenoe
Ballylegan
Lissava


N
2m


This study
Braadbaart and Poole 2008
Mean random reflectance (%Ro)
0
1
2
3
4
5
6
7
300
400
500
600
700
800
900
1000
1100
Temperature (oC)


y = -6e-8x3 + 0.0001x2 - 0.044x + 5.9035
R2 = 0.99
5
4
3
2
1
0
%Ro mean random
300
400
500
600
700
800
Charring Temperature (°C)

Site	Reflectance sample no	Feature No. and type	Charcoal	Bone	Bone colour	Mean reflect.	Min reflect.	Max reflect.	No. of readings	SD	Temp - average (°C)	Temp - max range (°C)	Absolute max per sample (°C)	Absolute max per feature (°C)
Templenoe	1	141 Grave pit	Maloideae C. Weber., *Quercus petraea* (Matt.) Liebl. *Quercus robur* L.	Human	White	1.00	0.67	1.60	50	0.22	**375-425**	325-510	510	510
	22		Fine dust >250 µm	Human	White	1.20	0.90	1.38	50	0.13	**390-440**	360-475	475	
	2	133 Grave pit	Maloideae C.Weber., *Fraxinus excelsior* L.	Human	Grey-white	1.07	0.67	1.41	50	0.18	**380-430**	325-475	475	**515**
	26		Fine dust >250 µm	Human	Grey-white	0.87	0.67	1.15	50	0.11	**360-410**	325-440	440	
	27		Fine dust >250 µm	Human	Grey-white	1.15	0.63	1.67	50	0.26	**385-430**	325-515	515	
	3	179 Grave pit	*Quercus petraea* (Matt.) Liebl. *Quercus robur* L.	Human	White	1.12	0.59	1.83	50	0.38	**385-430**	325-525	525	525
	4		Maloideae C. Weber sp.	Human	White	1.14	0.73	1.52	50	0.21	**385-430**	345-490	490	
	24		Fine dust >250 µm	Human	White	1.01	0.59	1.53	50	0.27	**375-425**	325-500	500	
	5		Maloideae C. Weber. & *Quercus petraea* (Matt.) Liebl. *Quercus robur* L.	Human	White	0.97	0.55	1.18	50	0.15	**370-415**	325-440	440	
	6	207 Grave pit	*Corylus avellana L.*, Maloideae C. Weber., *Ulmus glabra* Huds.	Human	Grey-white	0.96	0.56	1.49	50	0.21	**370-415**	325-490	490	490
	25		Fine dust >250 µm	Human	Grey-white	1.10	0.67	1.47	50	0.22	**385-430**	325-490	490	
	7	265 Grave pit	*Quercus petraea* (Matt.) Liebl. *Quercus robur* L. , Maloideae C. Weber.	Human	White	0.90	0.56	1.12	50	0.14	**360-410**	325-430	430	430
	8	33 Domestic pit	Maloideae C. Weber., *Corylus avellana* L, *Ilex aquifolium* L., *Prunus spinosa* L.	No bone		1.05	0.88	1.30	50	0.09	**375-425**	360-460	460	460
	9	193 Grave pit	*Quercus petraea* (Matt.) Liebl. *Quercus robur* L.	Human (older adult)	White	1.09	0.68	1.35	50	0.16	**380-430**	325-470	470	**500**
	19		Fine dust >250 µm	Human (older adult)	White	1.26	0.93	1.56	50	0.16	**390-450**	360-500	500	
	10	199 Possible pyre	*Quercus petraea* (Matt.) Liebl. *Quercus robur* L.	No bone		0.89	0.68	1.15	50	0.12	**360-410**	325-435	435	435
Ballylegan	11	446 Hearth	*Prunus Prunus avium* L. (L.) *Prunus padus* L., *Salix* L., *Alnus glutinosa* (L.) Gaertn., *Euonymous* L., Maloideae C. Weber., *Quercus petraea* (Matt.) Liebl. *Quercus robur* L.			0.97	0.48	1.48	50	0.20	**370-415**	325-480	480	480
Lissava	12	12 Trough	Maloideae C. Weber., *Quercus petraea* (Matt.) Liebl. *Quercus robur* L., *Fraxinus excelsior* L., *Ulmus glabra* Huds., *Corylus avellana* L., *Salix* L.			1.25	0.85	1.50	50	0.15	**390-450**	355-490	490	490

	Feature No. and type	Absolute max per feature containing bone (°C)	Absolute max temp. per feature - no bone (°C)
Templenoe	141 Grave pit	510	
	133 Grave pit	515	
	179 Grave pit	525	
	207 Grave pit	490	
	265 Grave pit	430	
	33 Domestic pit		460
	193 Grave pit	500	
	199 Possible pyre		435
Ballylegan	446 Hearth		480
Lissava	12 Trough		490

Temperature (°C)	Low/High %Ro	Maximum %Ro variation
300	0 - 1.2	1.2
350	0.26 - 1	0.83
400	0.6 - 2	1.4
450	1.05 - 1.4	0.35
500	1.39 - 2.5	1.81
550	2.1 - 2.6	0.5
600	2.65 - 3.85	1.2
700 *	3.15 - 4.9	1.75
800 *	3.71 - 5.4	1.69
900 *	4.95 - 6	1.05
1000 *	5.23 - 6.1	0.29
1100 *	5.91 - 6.2	0.26

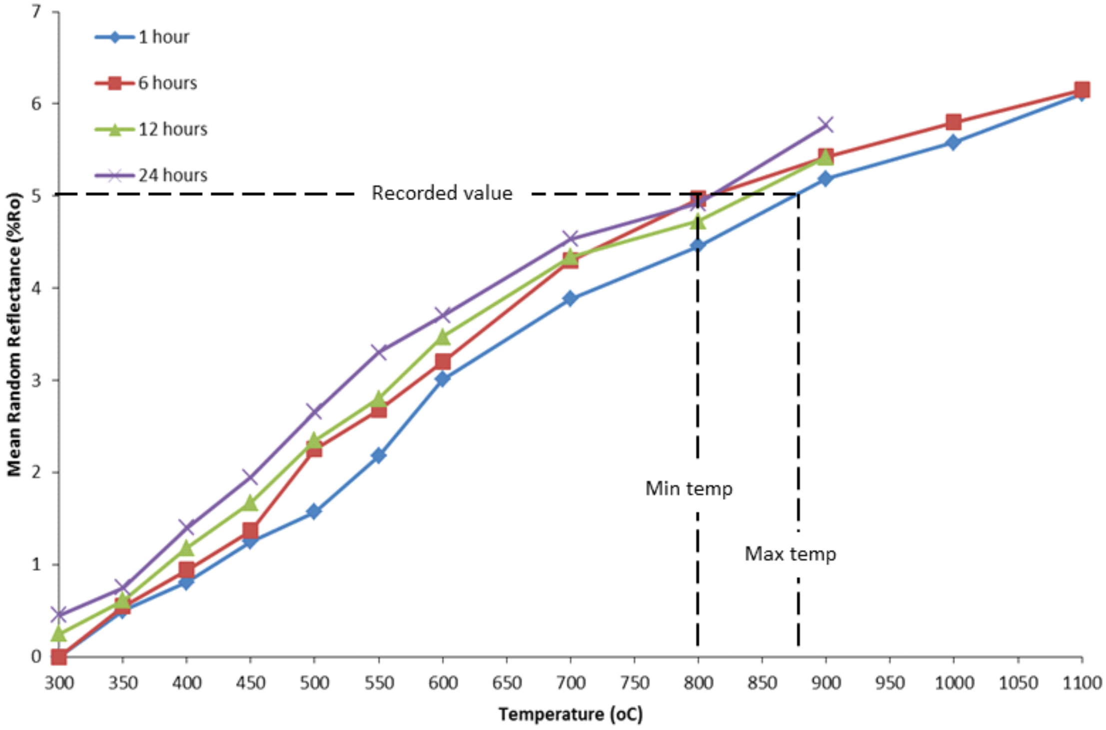
Overview of reflectance

Reflectance testing has been adopted in coal assaying where it is standardized and used to rank coal quality (see for example https://www.astm.org/Standards/D7708.htm). Charcoal also has an exceptional ability to reflect light when viewed using reflectance microscopy. The amount of light reflected is variable depending on the differential ordering of graphite-like phases within the charcoal itself. The charcoal's carbon atoms organize into an increasingly regular matrix over time. An analogous process is that of the less regular structure of graphite, progressing to the highly organized structure of diamond gradually over time, and with heat and pressure. In charcoal it has been demonstrated that this relates to the temperature of formation, whereby higher (absolute) formation temperatures result in higher charcoal reflectance (due to the more formal ordering of the carbon atoms). Cell wall fusion or homogenization may also play a role. This phenomenon occurs above 325 °C (Scott and Glasspool, 2005; McParland *et al.* 2007, 2009a, 2009b). Testing the reflectance of charcoal, (as for coal), requires collection and mounting of samples in resin. Transverse sections of each of the fragments of charcoal are embedded in polyester resin and polished to a highly smooth surface. In this study, two methods were employed, cold and hot set. The cold set method requires careful manual placement of charcoals at the base of a mould, followed by mixing and pouring in of two part epoxy resin, (without disturbing the sample), and curation (about 24 hours), before careful polishing. The automated hot epoxy method raises the materials to no more than 80° C. Resin tablets can be prepared in about 15 minutes from powdered epoxy, (Citopress hot press) and polishing can be more automated so as to polish multiple samples at once, thus reducing preparation time. At the Materials Laboratory at the University of Sydney, six samples were polished simultaneously, (Struers equipment, using increasingly fine grinding surfaces) allowing complete preparation of six samples in about two hours. Reflectance is measured using a reflecting microscope (in this case, the Nikon microphot microscope attached to Leica QWin image analysis software (Leica Image systems Ltd., 1997). Specimens are measured under Cargill immersion oil (refractive index, Ro, of 1.518 at 23 °C), using the x40 objective lens. Calibration is required, and in this study, the performance of the laser was calibrated against five standards: Spinel (Ro 0.393), YAG (Ro 0.929), GGG (Ro 1.7486), cubic zirconium (Ro 3.188) and silicon carbide (Ro 7.506).

Once calibrated, the laser light is bounced off the charcoal surface and its reflected value (Ro) recorded. Usually a number of measurements are made on one sample (50, or even 100), thus providing a range of Ro readings for one fragment of charcoal. If the charcoal concerned is very small (i.e. micro, rather than macro-sized), measurements in the 'one' sample may come from several small fragments. From the range of measurements for one sample, an average is calculated for that sample (Ro avge), and of course there are Ro min and Ro max measurements for that sample (as measured, but these may or may not represent actual minimum and maximum of the sample).

Reflectance calibration curves

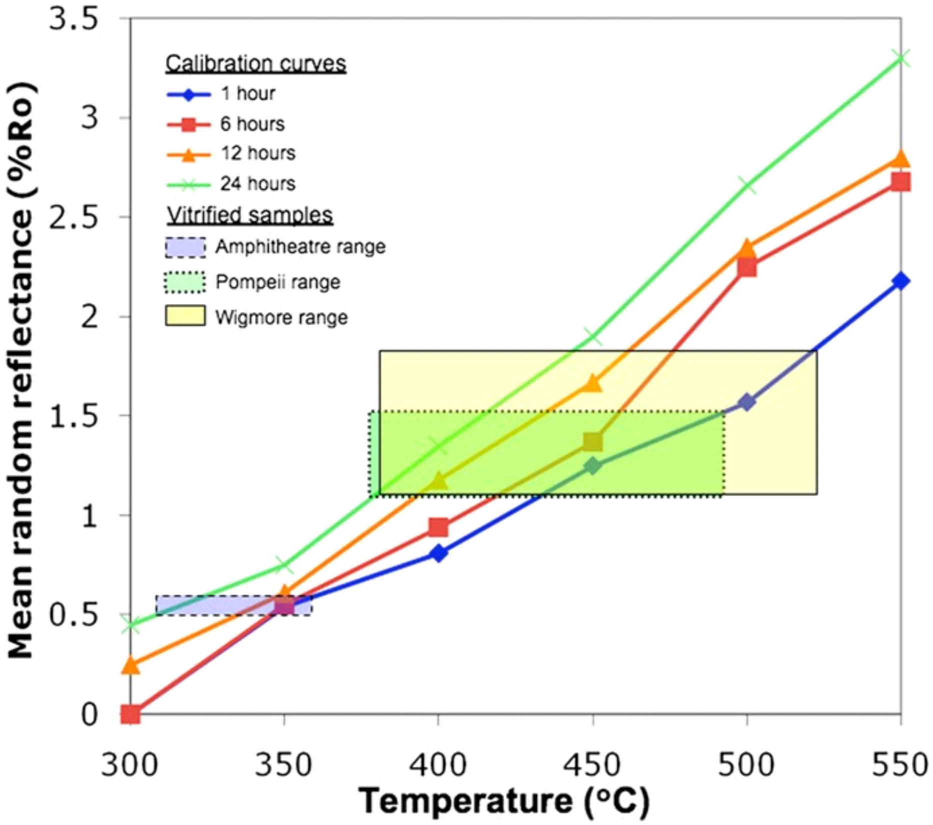
A number of researchers have charred woods of various types under varying (controlled) conditions, measuring the absolute temperature of charring, over various timeframes, and then subsequently measuring the reflectance of these modern samples, in order to produce a calibration curve that relates the temperatures observed with the measured reflectance values. McParland et al. (2009b) produced their calibration curve by plotting the reflectance results (y-axis) against the known temperature of formation (x-axis) (Fig. 1). The formation temperatures of samples with unknown charring temperature may then obtained from the curve using the measured reflectance value (on the y axis), and reading the associated temperature from the x-axis. In this study, the temperature calibration curve for oak created by McParland et al (2009) has been used since oak was the most common taxon in the cremation assemblages. Other curves for softer, harder, and/or more resinous woods read slightly differently. Temperature results of the archaeological material examined here were inferred using a 1, 6, 12 and 24 h calibration curve, i.e. the modern wood samples were charred and their temperatures measured – after 1 hour, 6 hours, 12, and 24 hours to create the calibration curve. The results are expressed as a range of temperatures e.g. a reflectance of 5%Ro would need to be charred at 900° C for one hour or 800° C for 24 h giving a range of 800-900 ° C. A natural outworking of these observations suggests that quick charring to a particular temperature, (i.e. in one hour), will produce a higher reflectance value than slower charring (e.g. 24 hours) to the same temperature. Since cremation of a full body takes about one day, we might infer that slower charring might have been occurring, however, flame temperatures fluctuate over the cremation process, for example, upon original ignition of clothes, and starter materials (brush woods, or potentially oils), and again when body fats reach ignition temperature. As stated in the main text, much of the body is burnt in a few hours, and even when cremation is 'completed,' some body parts may not have been burnt.



SUPPLEMENTARY FIGURE 1 Calibration graph of reflectance vs burn temperature (modified from McParland et al. 2009b) (Corresponds to figure 1 in main text).

The reflectance observed in charcoals is anisotropic in nature and therefore, the measurements taken will depend upon the position of the sample relative to the plane of polarisation of the laser light. Reflectance is reported either as maximum reflectance (R max) or random reflectance (%Ro). R max is measured by orientating the sample or polariser to provide the reflectance maximum (which takes time to carry out). %Ro (mean random reflectance) simply takes a mean from the sample (from a number of measurements taken at random). Samples in this case have not been aligned to find the Ro max and therefore will often be lower. Mean random reflectance is reported throughout this study, in that the sample is not orientated before measuring. There is still a minimum random reflectance value and a maximum random reflectance value, however, these should not be confused with true maximum reflectance (R max).

Generally, in coal samples, and in some other charcoal reflectance studies, calculating the average measurement is sufficient, however, given we are interested in the highest observable temperature as randomly measured (i.e. %Ro max), we ultimately refer to the highest random measurement taken for each sample. By way of further explanation, in a study examining the phenomenon of 'vitrification' in archaeological charcoal, (McParland *et al*, 2010), the reflectance ranges were measured for a variety of charcoals obtained from different charring/burning modalities (see Supplementary figure 2). Reflectance of wildfire charcoals is also now carried out to test differences in wildfire regimes, e.g. crown fires vs ground fires, the former being much hotter than the latter, (Hudspith *et al.,* 2015; Belcher and Hudspith, 2016).



SUPPLEMENTARY FIGURE 2. From McParland *et al.* 2010. Range of mean random reflectance (%Ro) of the vitrified areas of the archaeological fragments from three archaeological sites: Pompeii, Wigmore and Chester amphitheatre, plotted against the calibration curves of temperature vs. reflectance (for experimentally charred oak) in order to show the range in temperature of formation of the vitrified charcoals. Laboratory calibration charring data from McParland et al. (2009a).

Fire, temperature and fuel behavior (supplementary material 2)

A detailed understanding of how fire behaves may inform our understanding of fuel behavior during cremation, and its probable location after cremation is concluded. Much of our understanding about fire arises from studying wildfires, as well as forensic studies of modern fires where human are incinerated. This discussion draws on these sources, as well as limited works on ancient cremation.

The building of a cremation pyre will result in the provision of: the major fuel, timber logs possibly constructed in a pyramidal shape (with decreasing diameter); the secondary fuel (the body, especially the body fat); and the ignition fuels (which may be a range of products from kindling and shrubs to oils, we have little evidence for these from cremation sites). Bodies in modern and Roman cremations are normally placed on top of the pyre, in a shroud, or indeed in clothing (either of these provide more low ignition fuels). The major ignition fuels are placed around the base of, and potentially inside, (or in the case of oil, poured on to), the pyre, and ignition of these commences the burning process. The fire so started, must eventually transfer enough heat to the unburnt major fuel, the wood (Scott *et al.* 2014:302), and then also to the body; heat may be transferred by three different processes (Scott *et al.* 2014:303):

i) Convection: the transfer of heat by movement of a fluid (liquid or gas), including by actual flame contact. In a cremation, this is the natural movement of hot air and gaseous combustion products upwards, a process that will be further assisted by a draft. Convective heat is not visible to the naked eye.

ii) Radiation: transfer of heat in straight lines at the speed of light from hot particles of matter (solid, liquid, or gas) to cooler regions in the fuel layer, or its surroundings. In a modern open air cremation, this is felt from the visible flames, and will increase as the cremation proceeds. Uneven burning/spreading of flames will cause hot air turbulence, which may be significant and result in movement of burnt and partially burnt fuels.

iii) Conduction: the transfer of heat through matter from a region of high temperature to a region of lower temperature. Again, conduction may add to turbulence, and thus fuel movement within the pyre.

As the ignition fuels burn, white vapours are emitted around 100 °C (if the fuel is not completely dry), this is water vapour. Following this stage as the temperature rises, organic hydrocarbons vaporize and are emitted (blue smoke). A similar pattern occurs with the wood. More detailed information is provided in Braadbaart *et al.* (2012:844), and Harrison (2013). Once flaming combustion is established on a fuel surface, it reaches an equilibrium condition where its temperature stabilizes no matter what the temperatures of the flames above may be (Dehann, 2008: 5). For wood the surface temperature of the horizontal fuel is of the order of 350-400 °C. Radiant heat from flames above, and smouldering combustion in the surface are balanced against radiative and convective losses, and the vaporization of the organic volatiles. Wood fires produce a maximum flame temperature of 1027 °C (Dehann, 2008: 4), however, flame temperature varies with height from the fuel surface. A steady flame reaches a maximum temperature just above the fuel surface (800-900 °C) (Denhann, 2008:6). Fires are typically ‘turbulent’, with the surface of the burning fuel rising and falling, and the atmosphere ranging from reductive to occasionally oxidative. This living and changing nature of actual fire is very different to the conditions wrought by placing bone (or wood), in a laboratory oven at a specific temperature.

The best fuel in the body is the subcutaneous fat, which has an auto-ignition temperature of approximately 350 °C (Dehann 2008:9). Body fat does not smoulder and will only burn as a flame, and it requires a rigid porous ‘wick’ to maintain the flame. This wick can be charred wood, clothing, or even bone. Body fat produces an average temperature of about 800 °C, and turbulent flames (Dehann 2008:9). It contributes substantially to the burning process, evaporating body fluids, degrading, drying and finally burning skin, and muscle (which burns reluctantly).

Modern house fires, where hot gases and pyrolysis products collect in a room, eventually reach a ‘flashpoint’ where the fire suddenly ignites these products, and temperatures can reach 1000 °C, with post flashover temperatures as high as 1200 °C (Dehann 2008:11). Some Roman cremations were held ‘inside’ four walls (open to the sky), presumably to keep heat in, as substantial marble constructions have been excavated (e.g. the ustrina of Antininus Pius and that of Marcus Aurelius). In a prehistoric cremation, we have no knowledge of the positioning of regularly used pyre sites in this respect, but it cannot be discounted.