Methods for U-series dating of CaCO₃ crusts associated with Palaeolithic cave art and application to Iberian sites

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ABSTRACT

U-series dating is a precise and accurate geochronological tool which is widely applied to date secondary CaCO₃ formation, for example in speleothem based palaeoclimate research. It can also be employed to provide chronological constraints for archaeological sites which have a stratigraphic relationship with speleothem formations. We present in detail our methods to conduct precise and accurate U-Th dating of calcite crusts that formed on top of cave paintings. Our protocols allow the application of U-series measurements on small, thin calcite crusts covering cave art, which can be found in many sites, while taking care not to harm the art underneath. The method provides minimum ages for the covered art and, where possible, also maximum ages by dating the flowstone layer the art is painted on. We present dating results for crusts from two locality types in Spain, a typical cave environment (La Pasiega) and a more open, rock shelter type cave (Fuente del Trucho).

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1. Introduction

Cave art is found on almost every continent and, in addition to its bearing on the origins of art itself, it constitutes one of the few sources of archaeological information about symbolic behaviour and social interaction among early humans. It has been critical to the definition of chrono-cultural groupings and successions that form the methodological basis of archaeology, and continues to play a major role in our interpretations of the world views and behaviours of past cultures. Accurate chronological control of the emergence and/or execution of cave art is essential for understanding the development and phasing of specific artistic periods (often referred to as ‘cycles’), and the relationship of these to archaeologically recovered evidence of human occupation. But despite more than a century of study, cave art has proven to be one of the most difficult archaeological phenomena to date.

The chronology of cave art is often derived from contextual and stylistic evidence, usually assigned to a specific archaeological period on the basis of stylistic comparisons with portable art objects that have been recovered from dated archaeological deposits, and through the construction of a relative stratigraphy of motifs that are demonstrably superimposed on cave walls (Breuil, 1952; García-Diez and Ochoa, 2013; Leroi-Gourhan, 1965). While stylistic grouping may be useful to identify connections between human groups in different geographical regions, a ‘chronology’ based on this method is potentially undermined by the possibility that specific themes or styles could have been produced in different areas at different times, and also that different styles could have co-existed at the same time. In short, one simply cannot assume a straightforward development that allows one to isolate well-defined and
chronologically distinct artistic periods. Furthermore, since very few direct dates have been produced for cave art, assigning a date to a stylistic group runs the risk of circularity, whereby the age relies on comparison with other stylistic groups rather than on criteria that are independent of style (von Petzinger and Nowell, 2011). In some cases the association underpinning the ‘dating’ is based on archaeological finds lying on the surface (floor) of a cave or in strata exposed by archaeological excavation. For these at least, a degree of chronological control exists (i.e. finds can be dated, for example, by AMS radiocarbon measurement). However, the ‘dating’ of cave art based on the assumption that the art is coeval with dated material recovered from elsewhere in the same cave is not based on an unambiguous and demonstrable stratigraphic relationship, and hence does not constitute a direct, robust chronology.

Radiocarbon dating, restricted as it is to the dating of organic materials, can be used for the direct dating of organic binders and pigments. This has been applied to a variety of cave art motifs, mainly charcoal-based paintings, the result of which dates the production of the charcoal and thereby provides a maximum age for the painting. In order to interpret the $^{14}C$ age of charcoal as the age of the art, an assumption is necessary that this occurred meaningfully close to the time at which the charcoal was used to create the art (Archaologically, this constraint is destructive to the art itself). It is necessary to subsample material from the art itself. However, AMS $^{14}C$ dating of art from some of the key European cave art sites was a major step forward (e.g. Valladares et al. (2001)), but many European cave art motifs are engravings or were made with inorganic red ochre, yellow ochre and/or black manganese and thus are not suitable for $^{14}C$ dating. Even where suitable materials are available for radiocarbon dating, the understandable precedence of the conservation of the art necessitates that miniscule quantities of pigment are removed for dating in order to keep visible damage to an absolute minimum. Sample sizes are therefore rarely ideal, rendering the samples more prone to problems of contamination than the larger samples used for more routine measurement of archaeological samples. Even where a sample of sufficient size can be removed, it is not always clear that the date of the organic fraction is contemporaneous with the execution of the art, e.g. in the case of old wood/charcoal used to make a black pigment (Pettitt and Bahn, 2003). Thus, uncertainty remains in many cases, and additional methods for dating cave art are clearly required.

The U-series method cannot be used to date cave art directly, since it cannot be applied to pigments, but in certain cases it can be used to provide age constraints for the art through the dating of associated carbonate deposits. Schwarcz and Blackwell (1992) suggested U-series dating of calcite formations covering cave art as a means of providing minimum ages for the underlying art, but at the time methodological constraints — especially sample sizes needed for alpha spectrometric U-series measurements — hampered application of this method. The adoption of thermal ionization mass spectrometry (TIMS) (Edwards et al., 1987) significantly reduced the sample size required and allowed the first U-Th applications to cave art (Bischoff et al., 2003; Pike et al., 2005; Plagnes et al., 2003). A further reduction of sample sizes was possible following the adoption of inductively coupled mass spectrometry (ICP-MS) (Shen et al., 2002) and multi collector (MC) — ICP-MS (e.g. Hellstrom (2003)) for U-series analyses. Thus, today it is possible to employ U-Th methods to a much wider range of sites and motifs and thus avoid any potential reduction of the sample size required for precise U-series measurements, using tailored MC-ICPMS protocols (Hoffmann, 2008; Hoffmann et al., 2007). This enables the dating of very small carbonate samples scraped from CaCO$_3$ (calcite or aragonite) formations covering cave art. It has been successfully applied in a number of recent studies, e.g. by Pike et al. (2012) and Aubert et al. (2014). Here, we describe our methods in detail and present and discuss dating results for cave art in two different locality types, a typical cave environment (La Pasiega, Puente Viesgo, Spain) and a more open, rock shelter type environment (Fuente del Trucho, Huesca, Spain). Our results were obtained in three different laboratories with identical setups. The results published in Pike et al. (2012) were measured in the Bristol Isotope Group laboratory (Bristol, UK); those presented here were measured in the U-series laboratory of the CENIEH (Burgos, Spain) and the U-series laboratory of the MPI for Human Evolution (Leipzig, Germany).

2. General considerations

The conservation and protection of the precious and unique cave art obviously has highest priority and is the underlying principle of our work. It is essential to avoid any damage to the cave art, and to minimise our impact on the cave environment to ensure the preservation of this global heritage for future generations. Because of its fragility, we cooperate closely with the authorities and researchers responsible for the management and curation of the sites where we work; we seek permission based on a detailed rationale that is assessed by expert panels, and one or more delegates appointed by the relevant government agencies oversees our sampling process, which is recorded and subsequently outlined in publications.

In many cases, it is simply impossible to remove samples, either because a potential risk of damage to the art is too great or our rigorous sampling criteria discussed below cannot be fulfilled satisfactorily. In our approach, only CaCO$_3$ that can be removed without any impact on the art is considered appropriate for sampling. However, impact on the CaCO$_3$ which formed before or after the painting was done (which is core to the dating method) cannot be avoided. The impact nevertheless entails no damage to the cave art itself, and for this reason alone the method is preferable to the removal of art pigments for radiocarbon dating. In order to ensure stratigraphic integrity (i.e. that all the calcite sampled had formed on top of the targeted artistic motif) and avoid contamination of our dating sample with either pigment or cave wall, we do not remove all the CaCO$_3$ and thus do not expose the covered pigment to the cave atmosphere.

All these considerations constrain the analytical techniques employed for sample characterisation and U-series dating. For example, in most cases it is impossible to obtain detailed information about CaCO$_3$ fabrics or the stratigraphy of successive growth layers, all of which would require removing solid CaCO$_3$ pieces for detailed microscopy. Although such sampling has been described and applied elsewhere (Aubert et al., 2007, 2014), we do not support a strategy requiring the removal of chunks of calcite including the pigment layers. While this may have the advantage of providing access to calcite above and below the pigment, it is destructive to the art itself.

The dating of cave art is a multidisciplinary endeavour which requires a wide range of expertise ranging from Palaeolithic archaeology to scientific dating methods. A dating team should therefore include experts in Palaeolithic archaeology, cave art, speleogenesis, speleothems and dating/chronology. Each cave and its art is unique, and experts associated with specific sites need to be included in the planning and sampling process. The application of the method presented in this study is limited to motifs where unambiguous stratigraphy of the art and overlying CaCO$_3$ can be established. Generally, cave art made with pigments is more suitable than engravings, because, with the latter, it can be difficult to define the boundary between the engraved bedrock and the overlying calcite, especially where the bedrock was speleothem-covered prior to the execution of the engraved motif.