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Mineral deposition systems at rock art sites, Kimberley, Northern Australia — Field observations

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ABSTRACT

Mineral coatings, fringes, glazes and skins forming on the surfaces of sandstone rock shelters in Western Australia's Kimberley region offer the potential to provide datable materials to bracket ages of rock art motifs with which they are often spatially associated. These mineral deposition systems, which occur at the interface between the atmosphere and host rock, have never been characterised specifically and their overall formation mechanisms have yet to be completely established. This study serves to increase the understanding of complex processes behind the formation and long-term preservation potential of these mineral deposition systems. This is achieved by combining field observations with multiple mineralogical and geochemical characterisation techniques. Using both wet and dry season field observations and 94 mineral accretion samples collected from three different areas of the Kimberley, we identify four separate mineral deposition systems; polychrome fringes, dispersed wall coatings, floor glazes and silica skins. Detailed observations of the different characteristics of each deposition system are used to assess their suitability for the application of radiometric dating methods. Coherent internal stratigraphies are identified in polychrome fringe accretions, essential for the reliable application of uranium-series dating techniques, whilst floor glaze mineralogy, identified as dominated by carbon-bearing calcium oxalate minerals, provides radiocarbon dating opportunities. Consequently, this study provides a rigorous basis for establishing targeted sampling and analysis strategies essential for reliable and replicable rock art dating as well as having implications for rock art conservation.

1. Introduction

Kimberley rock shelters are often characterised by an array of minerals forming at the interface between the atmosphere and the rock surfaces. These shelters are most commonly formed in the region's vast outcrops of sandstone, predominantly the 1.8 Bya King Leopold and Warton units, and are generally exposed to the extreme seasonal fluctuations in rainfall and temperature. The characteristics of such accretions vary widely: from micron-thick silica skins (Watchman, 1992), to the centimetre-scale crusts observed in this study. Accretions display a wide range of inter-layered mineral groups forming on the quartzite and sandstone substrates. These accretions may comprise many layers of alternating mineral phases with no single process or source likely to be responsible for the formation of an accretion. However, our research has found that sulphates, oxalates and phosphates dominate, with much rarer occurrences of nitrate minerals. Preliminary results indicate that these accretions accumulate and persist over extensive time intervals, spanning tens of thousands of years and an improved understanding of their occurrence allows us to better understand the geochemical processes contributing to rock art's preservation and degradation.

Globally, mineral accretions have been identified as having a dateable relationship to rock art (Aubert et al., 2014; Chalmin et al., 2016; Russ et al., 1996; Watchman, 1990). In the Kimberley these formations also offer opportunities for the application of multiple absolute dating techniques to individual accretion layers, providing an important opportunity to generate bracketing age information for rock art painted on or engraved into the rock surface. This is particularly important in the Kimberley rock shelters, host to one of the world's richest concentrations of rock art, which exists in multiple 'traditions' or styles (Aubert, 2012). Current understandings of the upper age constraints on the production of rock art from both Europe and Indonesia are > 35 ka, demonstrating that the human capacity for 'art' most likely developed independently and yet coevally on either side of the Pleistocene Eurasian land mass (e.g., Aubert et al., 2007, 2014; Pike et al., 2012; Quiles et al., 2016; Valladas et al., 2001). Indeed, the human capacity for symbolic competence and expression – but not necessarily rock art as we currently understand it - seems to have originated in Africa as far back as 80–100,000 years ago (Tattersall, 2016).

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In contrast, despite evidence for human occupation in Australia > 50 ka (O'Connor and Fankhauser, 2001; Hamm et al., 2016; Hiscock, 2013; Hiscock et al., 2016; Roberts et al., 1990, 1994; Veth and O'Connor, 2013; Veth et al., 2017), and a very rich rock art record; a paucity of reliable dates hinders the production of a robust chronology for the rock art motifs and styles observed in particular regions (but see David et al., 2013; Roberts et al., 1997; Morwood et al., 1994; Morwood et al., 2010; Ross et al., 2016; Walsh, 2000; Watchman et al., 2005; Watchman, 1997; Watchman et al., 1997). Indeed, until recently, many researchers did not accept a Pleistocene age for rock art production (see Jones et al., 2016; Ross et al., 2016).

The Kimberley is home to what is arguably Australia's oldest dated rock art in the form of an ochre-sprayed limestone fragment recovered from ca. 41 ka archaeological deposit at Carpenter's Gap (O'Connor and Fankhauser, 2001). Furthermore, rock art continues to be produced in the Kimberley today (Akerman, 2014; Blundell and Woolagoodja, 2005; Chalarimeri, 2001; O'Connor et al., 2013). Unfortunately, despite significant advances in the dating techniques applied to rock art globally (Jones et al., 2016), determining direct and indirect ages of both rock engravings and rock paintings continues to present a challenge. The challenge in dating Kimberley rock art using some techniques such as uranium-series dating lies in the predominantly sandstone landscape, which lacks secondary calcium carbonate. In addition, the preferential use of ochre pigments precludes any straightforward application of well-established radiocarbon dating techniques directly to the rock art. Globally, mineral accretions have been identified as having a dateable relationship to rock art (Aubert et al., 2014; Chalmin et al., 2016; Russ et al., 1996; Watchman, 1990) and this study addresses the potential for generating bracketing age information for rock art painted on or engraved into the rock surface in the Kimberley using the mineral accretions observed.

Consequently, this study forms the first stage in developing and applying radiogenic dating techniques to non-calcite bearing mineral accretions and mud-wasp nests spatially associated with Kimberley rock art. We here report on a multi-year collaboration between Indigenous partners, archaeologists, earth scientists, pastoralists and philanthropists to better understand this world-class heritage. This project uses and builds on previous rock art dating initiatives and aims to produce a suite of reliable age brackets for a variety of rock art in different Kimberley regions. The dating work will help tether rock art to other data sets such as archaeological excavations, palaeoenvironmental reconstructions, and recorded ethnography, but its success is dependent upon a thorough assessment and understanding of the materials being dated.

1.1. Setting

North-west Australia's Kimberley region covers a vast and sparsely populated expanse of ~421,000 km^2, much of which is marked with tens of thousands of Aboriginal rock art sites (Aubert, 2012; Ross et al., 2016; Walsh, 2000; Welch, 2016). While the exact extent of the 'Kimberley' differs depending on whether geological, cultural or administrative boundaries are used, it encompasses the northern portion of Western Australia, bound onshore by the Northern Territory state border to the east, the Indian Ocean to the west, the Timor Sea to the north, and the desert regions to the south (Fig. 1). Average seasonal rainfall in the Kimberley varies widely across a north to south gradient and thus here, rainfall values have been calculated from Bureau of Meteorology stations located in relative proximity to many sample collection sites for this study. The wet season occurs between October and April, with average monthly rainfall exceeding 184 mm in some regions, and average maximum daily temperatures above 34 °C as measured at several Bureau of Meteorology stations between 1988 and 2016 (Stations 001025, 001019, 003032, Bureau of Meteorology, 2016). The dry season takes place between May and September, during which time the average monthly rainfall decreases to around 10 mm and maximum daily temperatures average around 32 °C (Stations 001025, 001019, 003032, Bureau of Meteorology, 2016).

Rainfall is mainly sourced from the Indian Ocean to the north-west during the wet season whereas air masses migrate from the south-east during the dry season. Bushfires are common during the drier months (Northern Australian Fire Information Website, 2016) and vegetation across the region is chiefly open savannah woodland (Legge et al., 2016). The Kimberley has experienced marked climatic change over time, so the current climatic regime should not be extrapolated into the past without reliable palaeoclimatic data sets. More stable are the flat lying, Palaeozoic sandstones and orthoquartzites that dominate the landscape, with most rock shelters occurring within the Warton and King Leopold Sandstone formations (Fig. 1). The sandstones are composed predominantly of fine-grained quartz matrix, with inclusions of tourmaline, muscovite, zircon and titanium-bearing minerals with silica cement resulting in essentially no remaining porosity (Fig. 2).

1.2. Fieldwork

Fieldwork was conducted between 2014 and 2017 and included both dry and wet season conditions. Fieldwork focused on observations of different mineral accretions present within rock shelters to identify similarities and differences in their composition, and to determine their spatial association with rock art. Kimberley rock art typically consists of rock paintings with some engravings and other anthropomorphic markings. Mineral accretions were sampled to test hypotheses relating to both the depositional processes involved in their precipitation, as well as the geochronological methods by which their age of formation might be constrained. Contextual samples were also collected from five shelters including floor sediments, animal droppings and surface pool water. Each sample from archaeological sites was approved – and often taken by - the relevant Traditional Owner. We also had a Section 16 permit issued by the WA Department of Aboriginal Affairs. Additional permissions, such as those from the WA Department of Parks and Wildlife, were obtained as required. Specific locations of rock art sites are not disclosed in this study in order to protect sites from unauthorised visitation and to respect the wishes of our indigenous partners. However, site localities are given a reference number that correlates to an access-controlled archaeological site catalogue held at The University of Western Australia's Centre for Rock Art Research + Management.

1.3. Defining the mineral systems

This study identifies four key mineral depositional systems observed from 94 samples at > 50 coastal and inland Kimberley rock shelters (Fig. 1). Extensive field observations and intensive mineralogical analysis of these systems provide a critical first stage of a broader project to date Kimberley rock art. The four mineral depositional systems (detailed in Section 3 and Fig. 3) are:

1) Polychrome fringes
2) Dispersed wall coatings
3) Floor glazes
4) Silica stalagmites and skins

Laboratory analyses confirm our field observations, showing each system to be mineralogically distinct. From this we infer that these systems have separate origins involving different biogenic or inorganic sources in the landscape, and the transport and deposition of each system occurs in discrete locations of the shelters. Importantly, each depositional system has different implications for radiometric dating. We suggest a thorough understanding of these systems is an essential basis for reliable dating studies. The detailed characterisation of each system and the identification of their dating potential in this study should serve as a guide to future dating projects aimed at targeting

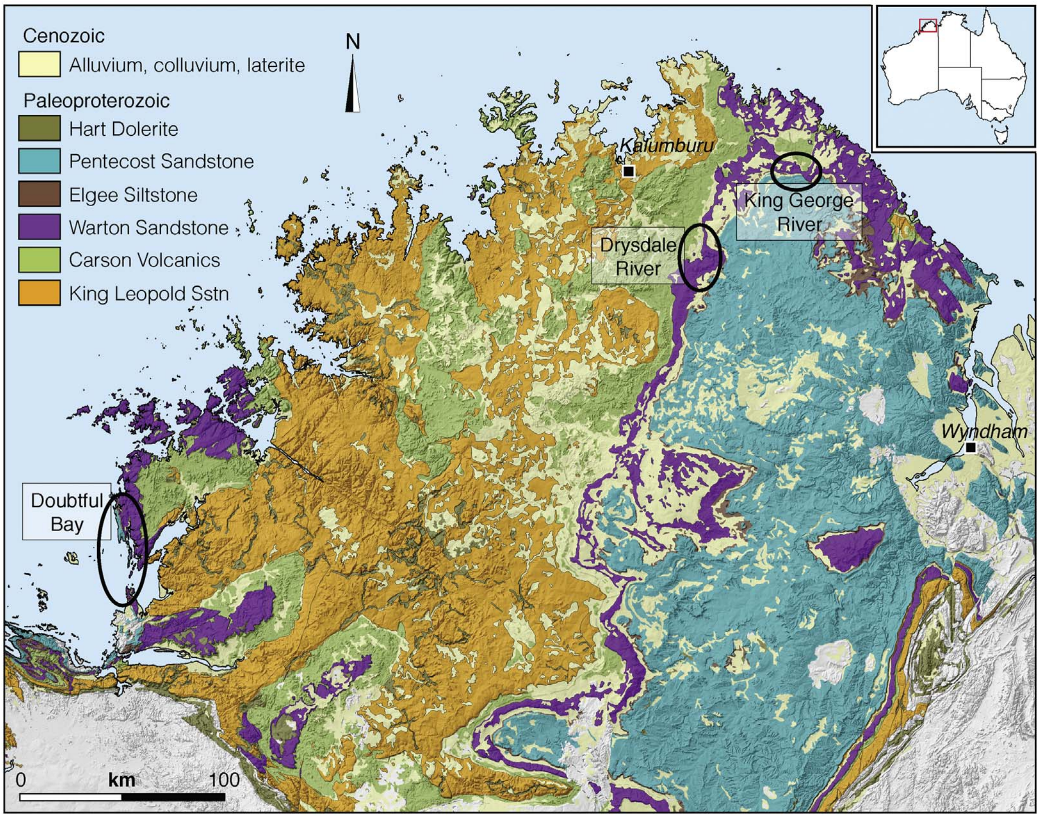

Fig. 1. Geological map of Western Australia's Kimberley with sampling areas. Samples were collected from coastal and inland sites near Doubtful Bay, the Drysdale River National Park and the King George River.

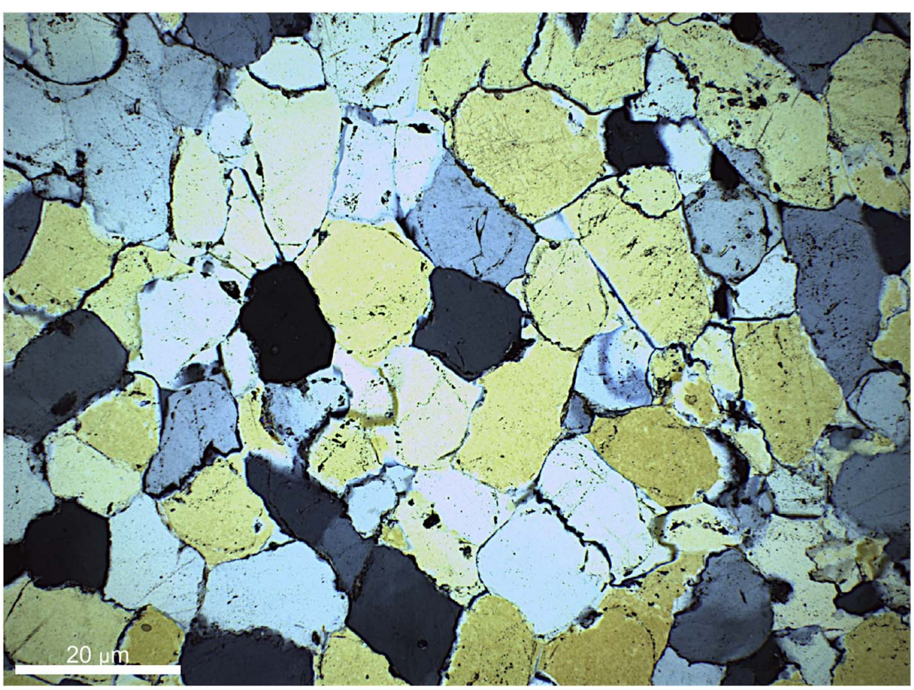

Fig. 2. Photomicrograph, taken under crossed polars, showing a thin section of the fine-grained Warton sandstone that hosts many Kimberley rock art shelters. The sandstone displays fine quartz grains with evidence of epitaxial overgrowths and silica cement resulting in minimal porosity. The sandstone also preserves and has inclusions of tourmaline, muscovite and titanium-bearing minerals.

mineral accretions spatially associated with rock art motifs. Each mineral depositional system also impacts the conservation of Kimberley rock art.

Characterising each of the four mineral depositional systems focused on identifying the internal stratigraphies and minerals present. Three techniques proved especially useful: X-ray diffraction, scanning electron microscopy, and laser-ablation trace element mapping.

2. Material and methods

Three primary mineralogical and geochemical characterisation methods were used to further assess the four identified mineral depositional systems, initially defined on their visual appearance and spatial occurrence in rock shelters. X-ray diffraction analysis was used to assess the different mineral phases present in the grouped accretion samples, whilst scanning electron microscopy identified minerals as being well crystallised. Finally, laser ablation trace element mapping

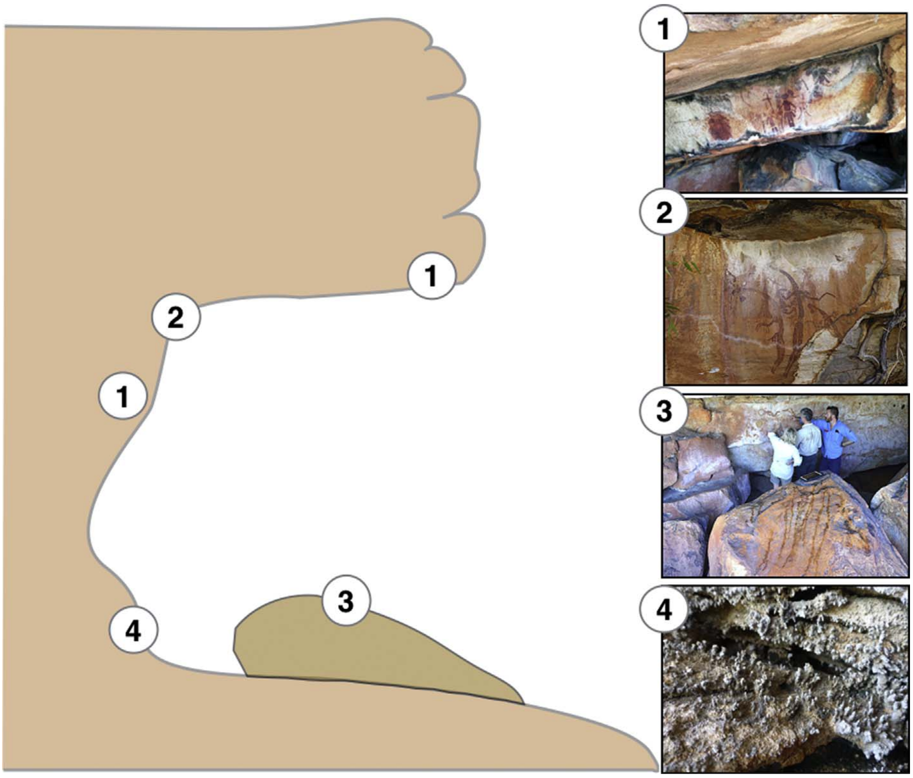

Fig. 3. Schematic model of a typical Kimberley rock art shelter displaying the dominant location of the four distinct mineral deposition systems: 1) Polychrome fringes 2) Dispersed wall coatings 3) Floor glazes 4) Silica stalagmites and skins.

examined the distribution of the different minerals within the internal stratigraphy of accretion samples, and identified phases that may prove suitable for geochronology (e.g., minerals with high uranium contents). Our results identified the presence of detailed internal micro-stratigraphies and a range of well crystallised sulphate, phosphate, silicate and oxalate minerals to be present in varying proportions dependent on the mineral system. Further details of both the data and methods for the mineral characterisation are presented in Green et al. (2017).

3. Results: mineral depositional systems

3.1. System 1) polychrome fringes

3.1.1. Occurrence and appearance

One of the most striking features of Kimberley rock shelters is a polychrome accretion 'fringe' often found seemingly to 'frame' panels of rock paintings (Fig. 4). These banded and visibly layered fringes occur on walls and along the outer edge of ceilings where areas of external water flow meet dry regions within the shelter.

Accretions display considerable variations in thickness, but range from < 0.1 mm at the gradient edge closest to the rock art, to ~1 cm thick at the outer margin. The surfaces of these accretions are often botryoidal in appearance with bisected nodules often revealing visible, continuous layering with each band varying in both colour and composition (Fig. 6 and Figures 1–8 in Green et al., 2017).

Although avocational rock art researcher Grahame Walsh speculated that rock paintings may have been preferentially positioned within these accretion 'frames' (Myers pers. comm. 2016), many accretions encroach into the painted panels suggesting that the 'framing' is coincidental (e.g., Fig. 4). Indeed, on many panels it is evident that reactions associated with leading edges of these fringes are destroying the rock art by partially to completely bleaching the iron oxide dominated pigments (e.g., Fig. 5D). An increased understanding of the accretion characteristics, formation processes and rates is crucial to both the production of reliable age information on the art and potential conservation and management strategies.

Fig. 4. Example of mineral deposition system 1, a mineral accretion fringe surrounding rock art at site 2195 in the Drysdale River National Park.

3.1.2. Mineralogical characterisation

Sulphate minerals are, by weight, the most abundant material in the mineral deposits of system 1, and occur as distinct bands in the multi-layered formations found on the thicker outer edges. A spectrum of compositions is preserved, with different bands dominated by sulphate species from distinct stages of the dehydration sequence ranging from gypsum ($CaSO_4.2H_2O$) to bassanite ($Ca(SO_4)(H_2O)_{0.5}$) and anhydrite ($Ca(SO_4)$). Polyhalite ($K_2Ca_2Mg(SO_4)_4{\cdot}2H_2O$) is also commonly present in the white accretion bands, averaging weight percentages of 20%, though rare examples with polyhalite contents exceeding 70% are also encountered (Green et al., 2017). Other detected sulphates include alunite $KAl_3(SO_4)_2(OH)_6$, syngenite ($K_2Ca(SO_4)_2{\cdot}H_2O$ and boussingaultite $(NH_4)_2Mg(SO_4)_2{\cdot}6(H_2O)$. Calcium and magnesium oxalate minerals weddellite ($Ca(C_2O_4){\cdot}2(H_2O)$), whewellite ($Ca(C_2O_4)(H_2O)$), and in rare cases glushinskite ($C_2MgO_4{\cdot}2H_2O$), have also been identified in 67% of the analysed polychrome accretion samples (45 samples). Although

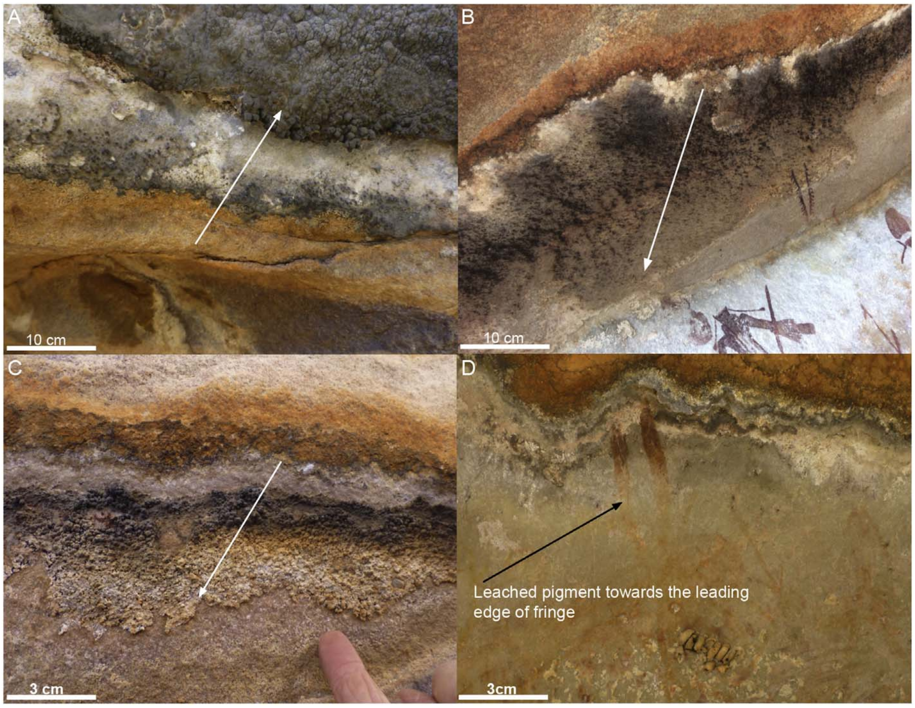

Fig. 5. A–C: Examples of the common colour sequence observed in polychrome fringes of mineral deposition system 1. D: An example of pigment leaching associated with a polychrome fringe. A and D are sites (BL005 and D06 respectively) located on the King George River, B and C (site DRY010) are in the Drysdale River National Park. White arrows indicate direction of water inflow.

oxalate minerals are not always present in system 1 accretions, when they do occur whewellite is the dominant phase. Indeed, the abundance of oxalate minerals is highly variable and XRD analysis has revealed that in some coastal accretions oxalates comprise almost the entire sample. Oxalate minerals have previously been described as occurring predominantly in black and pale-brown accretions found on the back wall of rock shelters (Watchman, 1991).

In contrast, this study has identified significant proportions of oxalate in white, brown and black accretions often occurring in discrete layers identified by laser ablation-ICP-MS mapping within bordering fringe deposits (Fig. 6 and Figs. 1–8 in Green et al., 2017). The occurrence of bands with high calcium and low sulphur content distinguishes oxalate-rich compositions from sulphate-dominated layers in these predominantly calcite-free environments. Phosphates, particularly newberyite $MgHPO_4(H_2O)_3$ and including taranakite $(K,NH_4)Al_3(PO_4)_3(OH)·9H_2O$, whitlockite $Ca_9·(Mg(PO_4)_6(PO_3OH)$, tinsleyite $KAl_2(PO_4)_2(OH)·2H_4O$, brushite $CaHPO_4(H_2O)_2$, varicite $AlPO_4(H_2O)_2$ and baricite $Mg_3(PO_4)_2(H_2O)_8$, have been identified in 49% (22 samples) of the polychrome fringes, in some cases dominating the accretion mineralogy (see Table 1 in Green et al., 2017). Although phosphates are identified in both black and white accretions, phosphate minerals are dominant in the white accretions. Laser ablation mapping also reveals discrete layering of phosphates and has allowed the identification of an important relationship between these layers and areas of elevated uranium concentration.

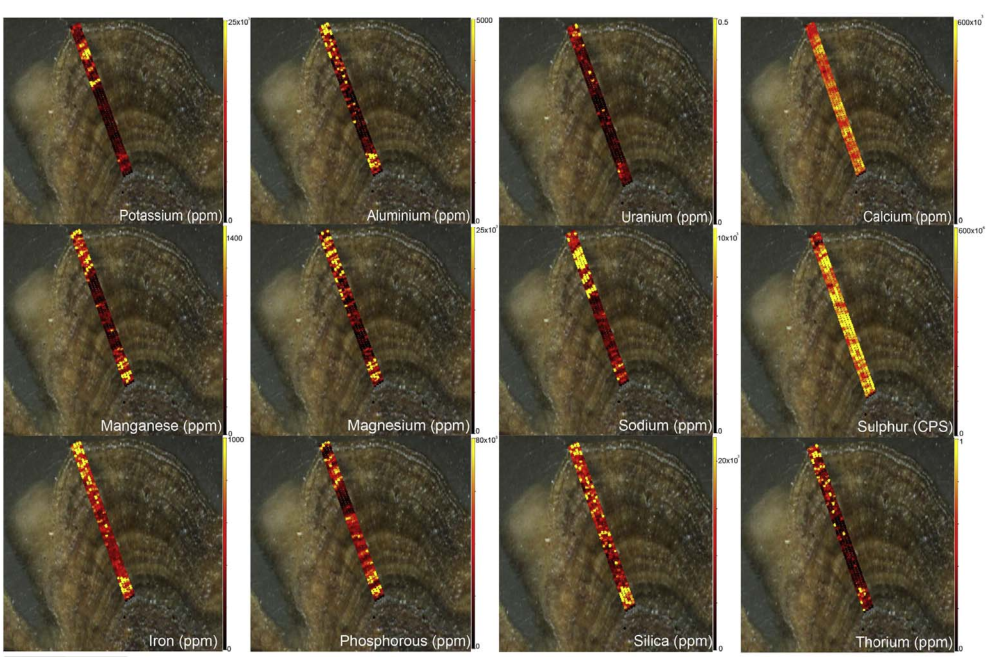

Fig. 6. Laser Ablation ICP-MS trace element maps for sample H221, a polychrome fringe accretion sampled at site DRY017. See Green et al., 2017 Fig. 8 for sample context photographs. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

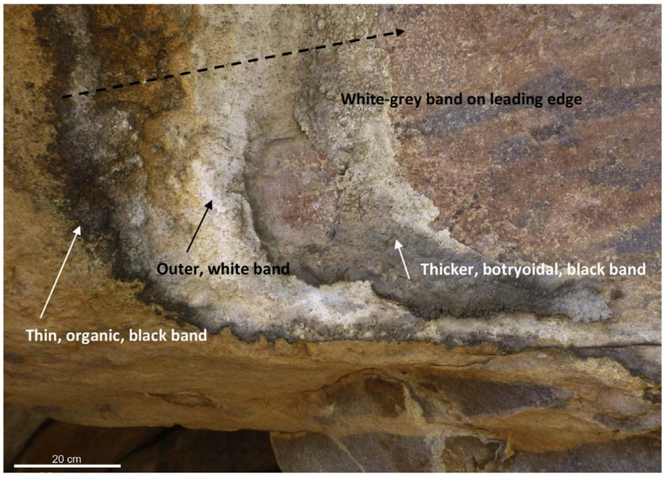

Fig. 7. Annotated example of the common appearance of polychrome fringes of mineral depositional system 1 in the Kimberley, site BL005, King George River. Dashed arrow indicates direction of water inflow.

3.1.3. Summary

Field observations have identified a consistent pattern (outermost thin black rim, white band, thicker black-brown band), in the colour and texture of the polychrome fringes between different rock shelters in our sample regions. The combination of XRD and LA-ICP-MS analysis has identified key phases that form the system 1 accretions and individual layers within them. Combining this information with other observations (e.g., thin-section descriptions) enables the following broad characterisation of mineral depositional system 1:

Outermost, thin, black rim: Microscopic examination of this accretion band identified the presence of crustose lichen. This band is consistently narrow (1–2 cm wide), black to dark green in colour, and displays a more distinct boundary on the inner edge and a graded boundary on the outer (Fig. 7).

White Band: This band commonly contains lower proportions of the less soluble sulphates such as gypsum and anhydrite and higher proportions of the hydrated sulphate polyhalite as well as magnesium sulphates such as boussingaultite. This white band also commonly contains oxalate in the form of both whewellite and glushinskite. Phosphate minerals such as taranakite and baricite have also been identified in minor quantities in this accretion type. Although wider, the white band is generally thinner in appearance than the black band, with a smoother, less botryoidal surface (e.g., Figs. 5B and 7). Consequently, XRD analysis is often dominated by the quartz signal as these accretions are more difficult to separate from the underlying quartzite.

Black/Brown Band: This band typically contains higher proportions of the less soluble sulphates than the other accretions described, with gypsum and anhydrite often dominating. Both whewellite and weddellite are commonly observed in the black, botryoidal domains within these accretions, often in comparable quantities to those observed in the white accretions. These may be accompanied by newberyite or other phosphate phases.

The common occurrence of oxalate in polychrome fringes is important as radiocarbon dating can be reliably applied to the carbon component of oxalate minerals. However, this reliability requires the age of the carbon in the mineral to be contemporaneous with the crust from which it was sampled (Watchman et al., 2005). Consequently, it is important that the origin of the oxalate is investigated in dating studies. The observation of a relationship between phosphorous minerals and uranium content in discrete layers within polychrome fringes is also, significant, providing a potential new approach to the application of uranium series dating methods to phosphate-bearing accretions.

Fig. 8. Example of mineral deposition system 2, 'Dispersed Wall Coatings', site 0343, King George River.

3.2. System 2) dispersed wall coatings

3.2.1. Occurrence and appearance

At many rock shelters rock art occurs close to bedding planes, and is typically underneath or sometimes on top of a thin, white 'wash' or coating of mineral aggregates. This coating seems to emanate from horizontal bedding planes that define the wall-ceiling boundary. Coatings are clearly controlled by the flow of water as the mineral deposits appear to seep down over the rock art, often accumulating in depressions in the rock surface (Fig. 8). In relatively rare instances where the accretion accumulates in a depression or ledge on the wall, thicker, often discoloured examples of this system may occur. Unlike other accretions observed in the Kimberley, coatings generally lack layering and commonly occur as a thin, dull coating found both underlying and overlying paintings (Figs. 8, 9 and 10).

Coatings range from chalky to crystalline in texture and predominantly white to cream in colour with yellow to pink tones observed at some sites. Where they are associated with rock art they gradually obscure the art, and in some cases, appear to react with the pigments producing changes, and sometimes gradients, in the original colouration. In rarer instances the white wall coating has been observed to replace or precisely over-coat the original pigment, resulting in paintings that are partially white in appearance (Fig. 9).

Fig. 9. Example of mineral deposition system 2, 'Dispersed Wall Coatings', replacing rock art pigment, site (KGRA004-A), King George River. Black arrow indicates areas of replaced pigment.

Fig. 10. A–D Examples of the dispersed wall coatings of mineral deposition system 2, commonly obscuring the rock art (A (site DRY00597), C (site D53), D (site D44) but in some cases providing an effective backdrop (B, site DRY006)). Drysdale River National Park. Also, in some cases the dispersed coating has completely covered and in some instances, partially replaced, mud wasp nest stumps, leaving behind partially mineralised pseudomorphs (Fig. 11).

In most cases the form of the original rock art motif can still be seen, but the thickest coatings sometimes completely obscure the art (e.g., Fig. 10A and C). These dispersed wall coatings have been recorded in shelters at both coastal and inland locations.

Also, in some cases the dispersed wall coating has completely covered and in some instances, partially replaced, mud wasp nest stumps, leaving behind partially mineralised pseudomorphs (Fig. 11). The mineralisation of mud-wasp nests is dependent on their location relative to the mineral systems identified in this study and therefore they do not represent an independent formation process.

Frequently, the coating will drape down the rock face, thinning towards its outer fringe. These coatings are rarely seen away from bedding planes or joints in the rock, and are more commonly observed on vertical walls than on shelter ceilings, suggesting a shared formation mechanism. Often the coatings are only sufficiently thick to surround the quartz grains of the host sandstone. Their chalky-crystalline nature means they are difficult to sample as anything other than small, powdery fragments. It is important to distinguish these coatings from white 'back-drops' of pigment sometimes utilised by artists to 'prime' the rock surface to bear rock art. These white painted back-drops are almost exclusively associated with Wanjina/Wandjina rock art (see Blundell and Woolagoodja, 2005). There are also often identifiable paint spatter marks from the production of these white paint backdrops that distinguished them from the dispersed mineral coatings, which are generally much more tenacious, lack brush marks and are also usually less continuous in their coverage of the rock surface.

Fig. 11. Mud wasp nest stumps mineralised by a dispersed wall coating, Doubtful Bay.

Mineralogical analysis of dispersed wall coatings in this study has identified a general lack of carbon (oxalate) and uranium (phosphate) bearing minerals conducive to geochronological methods (Fig. 12 and Figs. 17–22 in Green et al., 2017). LA-ICP-MS analysis also identified an absence of clear, continuous internal layering, further hindering the application of dating methods by preventing testing of closed system conditions and age replication (i.e. age estimates get 'younger' towards the surface of the sample, while ages along a layer are the same).

However, rare occurrences of particularly high uranium and thorium levels, identified by LA-ICP-MS have been found exclusively in these white wall coatings (Green et al., 2017). Consequently, sampling for geochronological work should target those rare occurrences of relatively thick accumulations of dispersed wall coatings, that may display internal layering allowing for the potential application of dating techniques. These have often been found to occur on ledges and crevices on shelter walls, allowing the usually thin, dispersed wall material to accumulate (Green et al., 2017). With the destructive impact these coatings have on the rock art (see below), understanding both the rates and processes involved in their formation is important in the consideration of future rock art conservation strategies.

3.2.2. Mineralogical characterisation

Mineralogical analysis of 25 dispersed wall coatings have revealed a distinctive mineral assemblage that differs from that of the other mineralogical deposition systems, notably, polychrome fringes and floor glazes. Hydrated sulphates such as polyhalite $K_2Ca_2Mg(SO_4)_4{\cdot}2H_2O$ are common with some samples almost entirely composed of this mineral. Rarer sulphate minerals such as alunite $(KAl_3(SO_4)_2(OH)_6$, an aluminum, potassium sulphate, are also observed in minor ($< 15\%$) amounts in dispersed wall coatings, possibly associated with mineralised mud wasp nests related to guano deposits in sedimentary

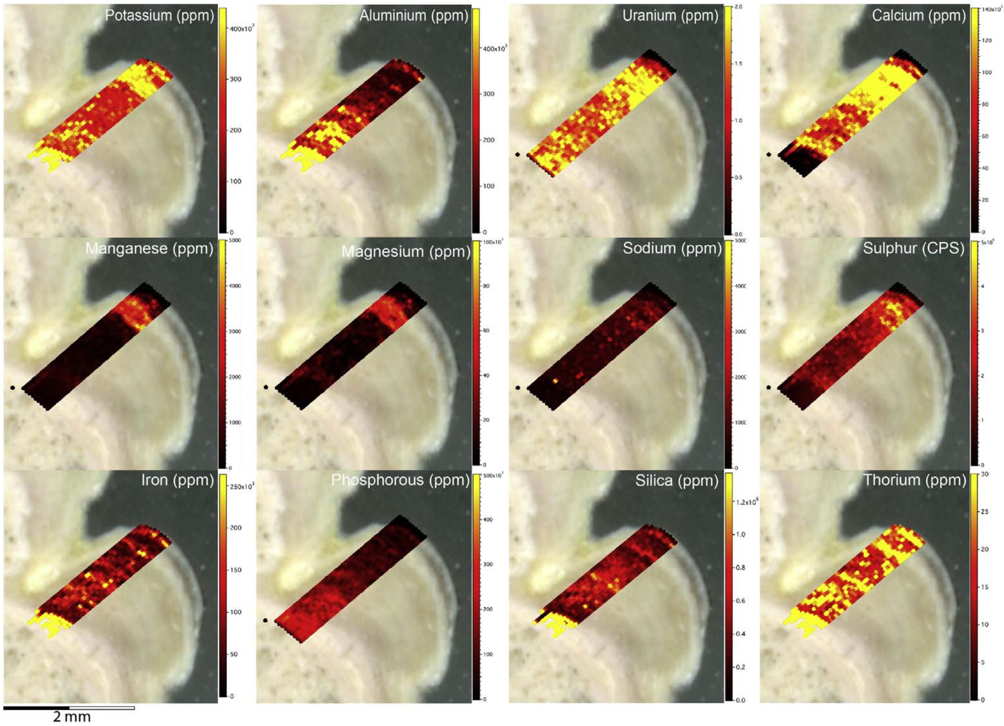

Fig. 12. Laser Ablation ICP-MS trace element maps for sample H040, a dispersed wall coating accretion sampled at site DRY010. See Green et al., 2017 Fig. 19 for sample context photographs. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

environments (Hill and Forti, 2004a; Jambor and Roberts, 1999). Alunite has also been identified in silica stalagmites observed in some Kimberley shelters, discussed below.

SEM-EDS results in dispersed wall coating samples such as K12–40 suggest the presence of colourless to white minerals; potassium-calcium sulphate syngenite $K_2Ca(SO_4)_2{\cdot}(H_2O)$ and niter KNO_3 (see Fig. 28 in Green et al., 2017). These minerals are often reported from cave environments as massive encrustations and efflorescent growths precipitating from solutions containing alkali potassium and nitrate (Anthony et al., 2016). Known to occur in animal dropping deposits (Pogson et al., 2014), these minerals have been identified in the dispersed wall coatings from sites in the inland Kimberley. During our fieldwork bats and wallabies were observed perching in crevices and guano covered rock faces in some rock shelters.

Phosphate minerals are rarely observed in the dispersed wall coatings but have been identified in significant quantities in some mineralised mud wasp nest samples using XRD methods. Phosphates taranakite $(K,Na)_3(Al,Fe^{3+})_5(PO_4)_2(HPO_4)_6{\cdot}18H_2O$, aluminum ammonium hydroxide phosphate hydrate $Al_2(NH_4)(OH)(PO_4)_2(H_2O)_2$ and leucophosphite $KFe_2(OH)(PO_4)_2(H_2O)_2$ have been identified in these samples, with the origin of most cave phosphate minerals attributed to guano (Hill and Forti, 2004a). However, wood ash has been found to contain up to 24% phosphate (Karkanas et al., 2000), providing a further potential phosphate source in a region that is particularly prone to extensive and seasonal fire events (Northern Australian Fire Information Website, 2016). Many rock shelters were also habitation sites (as evidenced by *in situ* cultural material and anthropogenic features and site modifications), where people likely brought wood to make fires for a variety of purposes.

3.2.3. Summary

Mineralogical analysis of the thin white to cream-coloured dispersed wall coatings observed in many Kimberley rock art shelters has identified a range of relatively rare and hydrated sulphate minerals precipitating alongside smaller proportions of potassium nitrate and chloride. Typically originating from the bedding plane interface of the shelter wall and ceiling, these coatings can be attributed to a combination of evaporitic sulphate, originating in local rainwater with potential contribution from sulphides in the host sandstone, and biological material. It is proposed here that on reaching the rock surface the concentration of this solution by evaporation of the thin water film in the desiccating environment of an exposed rock shelter would lead to supersaturation and precipitation of the observed minerals.

The association of many of the observed minerals with guano is consistent with the dominant location of these coatings at sites where birds and bats have been seen to roost. However, further sources, not previously considered, include the organic material contained in the large mud wasp nest accumulations frequently found in these locations; also, ash - either blown or washed into shelters following anthropogenic and natural fire events.

3.3. System 3) floor glazes

3.3.1. Occurrence and appearance

Fallen blocks and boulders are commonly found on the floors of rock art shelters. Frequently these are ‘glazed’ with accretions displaying very different characteristics to those observed on the rock walls. These coatings are always found on horizontal or sloping surfaces on the floors of shelters or on loose boulders (Fig. 13). They are rarely found on rock walls and are entirely absent from ceilings. The glazes are often found in association with sites of persistent animal urination and traffic. In rarer instances, these floor glazes may be associated with lichen growth. Accretions are often darker in colour compared with those of systems 1 and 2, and range from brown to black with a distinctive smooth and mostly shiny surface with a polished appearance. This appearance suggests urine and subsequent polishing by animal movement in shelters are the main vectors producing these glazes.

Glazes observed in this study are mostly 1–5 mm thick. Examination in hand sample and thin section, shows that they are often internally layered with very thin (sub-mm scale), uniform layering (Fig. 14 and see Figs. 9–16 in Green et al., 2017). They are usually well-developed over large areas of fallen slabs and shelter floors but are also occasionally found in small hollows on wall ledges and in narrow partings at the back of shelters, away from water flow. In places where these glazes are observed to have interacted with flowing water, they show evidence of dissolution. Glazes often exhibit scratch marks and parallel grooves

Fig. 13. Example of mineral deposition system 3, a 'floor glaze' with fresh animal urine, from a rock shelter (site D47) in the Drysdale River National Park. Inset; Camera traps that have been deployed at some sites have recorded the presence of rock wallabies using surfaces such as that shown here.

which may indicate human or other animal activity. In some cases, glazes bear clear anthropogenic rock markings and even rock engravings (Fig. 15).

3.3.2. Mineralogical characterisation

Mineralogical analysis of floor glazes shows that despite their disparate appearance, their mineralogy is like that of mineral depositional system 1. For example, the same dominant mineral groups (sulphates, oxalates and phosphates) observed in the thicker, botryoidal accretions seen in polychrome fringe deposits of system 1, have the same dominant mineral groups as those identified in the floor glazes. However, the proportions of mineral groups in each case indicate different deposition mechanisms. The advantage of the similarities observed between two mineral deposition systems (glazes and polychrome fringes) is that radiogenic dating techniques can potentially be applied to both deposit types, providing a wider scope for bracketing associated rock art. Whewellite ($CaCO{\cdot}H_2O$), a monohydrated calcium oxalate and less commonly, weddellite ($CaC_2O_4{\cdot}2H_2O$), a dehydrated calcium oxalate salt, have been identified as comprising substantial portions of layered mineral crusts collected from both coastal and inland Kimberley sites (see Table 1 in Green et al., 2017). Oxalate is found in much more significant proportions in all analysed floor glazes (7 samples) than on wall coatings. Quantitative estimates of mineral proportions by XRD analysis have identified some floor glazes to be composed predominantly of oxalate ($>$ 60%). The occurrence of oxalate minerals in Kimberley mineral depositional system 3 alongside evidence for elevated uranium concentrations in discrete layers (Fig. 14 and see Figs. 14–19 in Green et al., 2017), is particularly important as it provides a potentially dateable medium by which minimum or maximum age estimates may be provided for any associated engravings and anthropogenic rock markings on the varnished rock blocks (Fig. 15A and D; see also Watchman, 2000).

In addition, floor glazes associated with rock paintings may be used to bracket that rock art in terms of dating the slab fall. Thus, either rock art-bearing rock that has fallen, and/or rock art painted in the resultant flake scar, can be constrained by such dating. Radiocarbon dating can be applied to the carbon component of both weddellite and whewellite if that carbon can be proven to be contemporaneous with the crust from which it was sampled. Calcium sulphates gypsum ($CaSO_4.2H_2O$) and anhydrite ($CaSO_4$) and magnesium phosphate newberyite ($MgHPO_4(H_2O)_3$) have also been found in low quantities in the floor glazes. Laser ablation ICP-MS elemental maps have identified

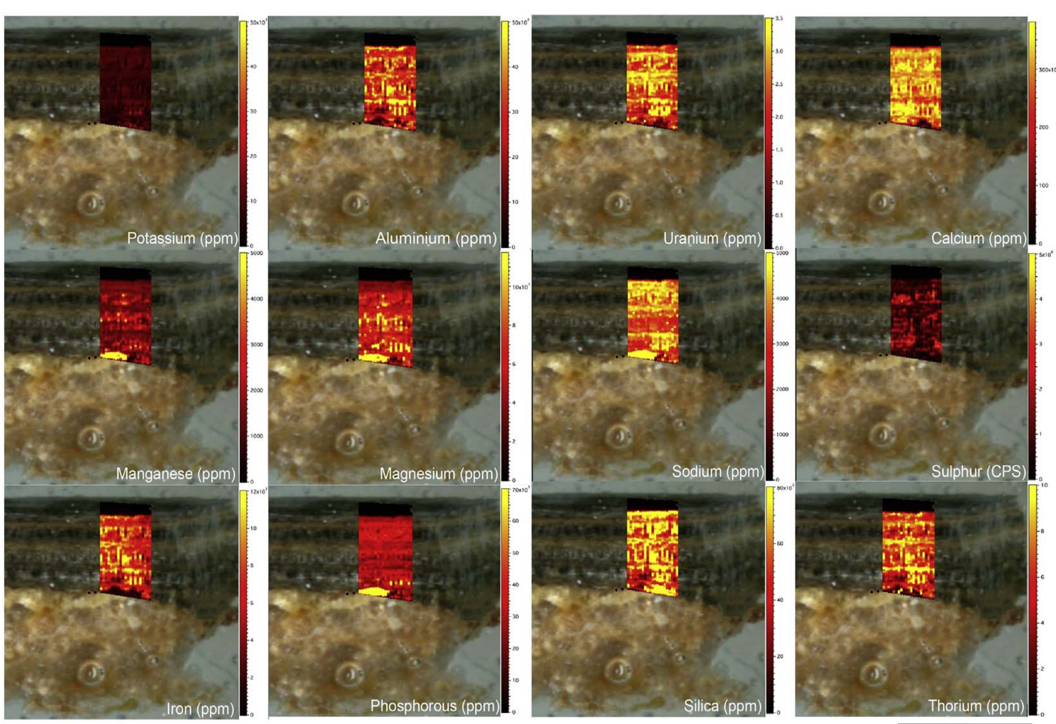

Fig. 14. Laser Ablation ICP-MS trace element maps for sample H076, a floor glaze accretion sampled at site DRY013. See Green et al., 2017 Fig. 15 for sample context photographs. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Fig. 15. Examples of mineral deposition system 3. A: Floor glaze with anthropogenic rock markings covering a boulder at the shelter entrance. B–C: Glazes are predominantly dark brown in colour, with smooth, shiny surfaces. D: Thick floor glaze covering a boulder with anthropogenic rock markings (indicated by the black arrow). Drysdale River National Park. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

continuous discrete layering, with some bands containing elevated concentrations of uranium, providing potential for the application of multiple radiometric dating techniques to these deposits.

3.3.3. Summary

Many shelters host boulders, rock outcrops and fallen slabs 'glazed' by thin, dark brown-black, shiny and smooth mineral deposits, covering relatively large surfaces (up to 1 m). Floor glazes often occur in areas accessible to small animals and away from water pathways. Recently deposited patches of animal urine often cover the varnished areas (e.g., Fig. 13), strongly suggesting that this plus subsequent 'polishing' through animal movements in rock shelters, are the dominant source of these glazes. Cognate research in southern Africa shows similar glazes produced by rock hyrax urination (Carr et al., 2010). Mineralogical examination of the glazes has identified high proportions of the calcium oxalates whewellite and weddellite, providing potential opportunities for the application of radiocarbon dating methods to those associated with significant rock slab falls or engravings. In addition, elevated uranium concentrations in the micro-scale layering observed in these varnishes provides a further independent opportunity to constrain the ages of associated rock art.

3.4. System 4) silica stalagmites and skins

3.4.1. Occurrence and appearance

Some Kimberley rock shelters display a thin and often transparent skin, which appears to coat the host quartz grains. These skins are often only discernible by the slight sheen they give the underlying rock surface and in some cases, rock art pigment (Fig. 16).

These skins are generally amorphous and uniform in coverage and are usually < 1 mm thick. In some locations, silica skins reduce the colour tone of painted motifs. However, it has been speculated that their occurrence aids rock art preservation, 'binding' pigment to the substrate (Watchman, 1990) and providing a layer resistant to chemical weathering. In the porous sandstone, the silica skin precipitates in the pore spaces. By filling these pore spaces and other depressions where mineral depositional system 2 accretions might have otherwise accumulated, these silica skins appear to prevent the often-destructive precipitation of dispersed wall coatings (e.g., Fig. 8). Unfortunately, the

Fig. 16. Example of mineral deposition system 4, a silica skin 'coating' a rock art panel at site KGRB002 in the King George River region.

Fig. 17. Examples of mineral deposition system 4. A: Here a silica skin 'coating' has developed into a thicker accretion displaying an undulating, nodular surface with fields of small silica 'stalagmites'. B: Thicker, silica skin coating. C–D: Silica stalagmites forming on ledges and 'wind passages' within a shelter (site BL005) King George River region.

stresses created by this precipitation can lead to the exfoliation of the outer rock surface and localised peeling of the silica skin resulting in the removal of associated pigment. For this reason, conservation strategies aimed at exploiting the observed benefits of these silica skins have so far proven unsuccessful.

At some shelters, silica skins have developed into a thicker (1–3 mm) grey-coloured accretion and display an undulating, wrinkled or in some cases, nodular surface quite different in appearance to the thinner, uniform skins (e.g., Fig. 17A and B). These accretions appear to be related to, but distinct from, the commonly observed 'silica stalagmites' often found in air passages or below rock ledges in Kimberley rock shelters, as shown in Fig. 17C and D. Silica skins are difficult to remove as intact pieces, sufficient in size for cross sectional analysis using LA-ICP-MS trace element mapping without causing significant damage to the rock art panel. Consequently, there are no example LA-ICP-MS maps for this mineral depositional system.

3.4.2. Mineralogical characterisation

Whilst silica skins are composed exclusively of amorphous silica, silica stalagmites from both coastal and inland sites contain other components. In three of the five silica stalagmites sampled both on the coast and inland, silica is the major constituent. However, two inland samples contain gypsum as the dominant phase with minor components of both quartz and in sample K12–28; whewellite. In the other inland example, alunite was identified as the dominant mineral alongside subordinate quantities of gypsum and quartz (see Table 1 in Green et al., 2017). Silica stalagmites generally accumulate in crevices and areas of air through flow in shelters and SEM imaging has revealed a crown dominated by well crystallised gypsum whilst the 'stem' is generally composed of silica (Fig. 18).

Sedimentary alunite has also been identified in silica 'stalagmites' in sandstone rock shelters in New South Wales, Australia, where its formation was linked to the reaction of sulphuric acid, concentrated in evaporating drip waters, on illite, smectite and kaolinite clay materials (Wray, 2011). Kimberley silica skins frequently show a reddish-brown colouration, often partially obscuring the art underneath. It is presumed this discolouration is due to the presence of iron oxides, perhaps from wind-blown dust, although the abundance of these are extremely low as they are below the detection limits of SEM energy dispersive x-ray analysis.

3.4.3. Summary

Although silica skins may play an important role in preserving Kimberley rock art at some shelters by seemingly securing the pigment into the sandstone substrate, they can also be an agent of destruction. The skins have also not proved particularly useful for dating (contra Aubert, 2012). By applying radiocarbon dating methods to organic material trapped within the silica skin, previous work has attempted to provide bracketing age constraints on associated motifs in the Kimberley (Watchman, 1996). This approach has been criticised because it requires the bulk sampling of the carbon bearing materials held in these skins and is unable to discriminate between different organic compounds, which impacts the reliability of the interpretation (Bednarik, 2002). The application of uranium-series methods to these skins is also unfeasible as they are usually so thin it is difficult to remove intact fragments, sufficient for LA-ICP-MS trace element screening, without causing significant damage to the associated rock art motif. Without an internal stratigraphy, discrete layering cannot be targeted to repeat ages or test for closed system conditions, factors essential in producing a robust and reliable chronology.

4. Conclusion

Rock shelters in north-west Australia's Kimberley region demonstrate at least four mineral deposition systems that often display close spatial relationships with the region's rich and varied Aboriginal rock art. The four different systems contain varying proportions of a broadly similar range of minerals dominated by sulphates, phosphates and

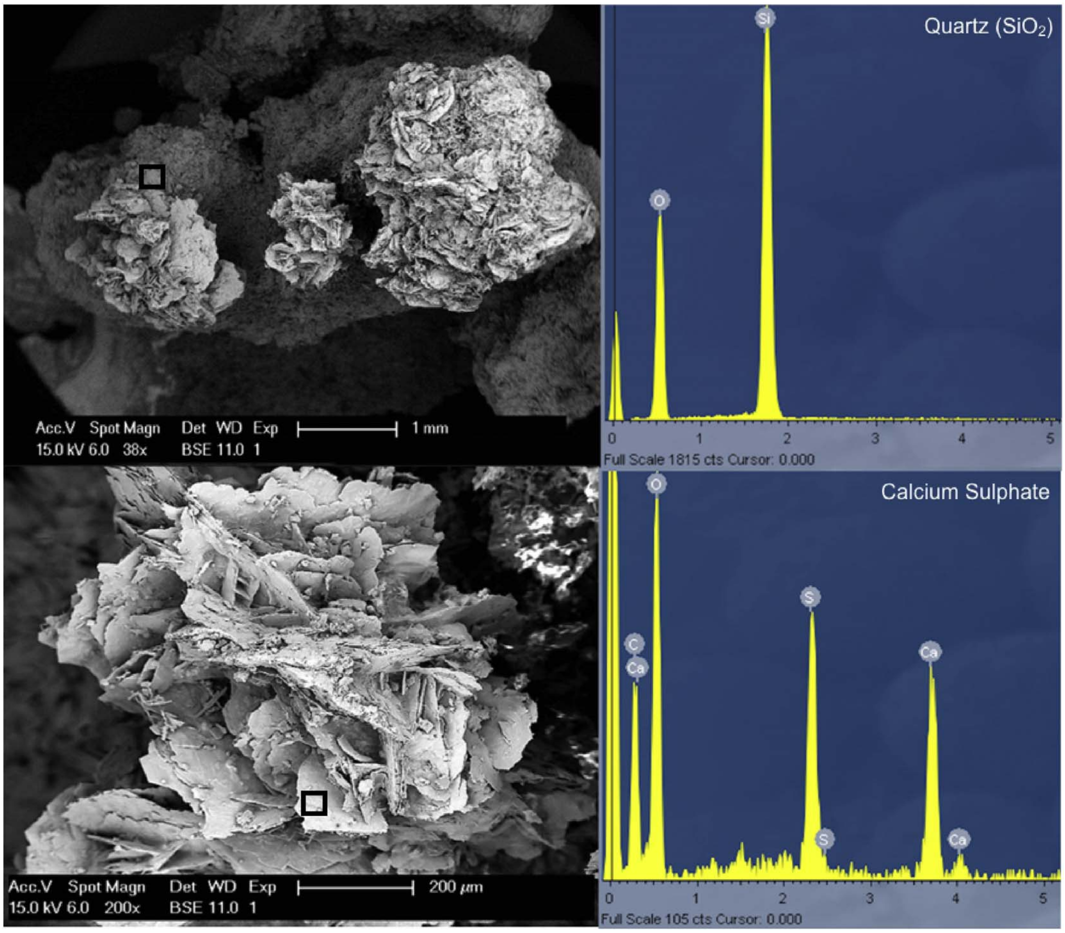

Fig. 18. SEM images of a silica stalagmite collected in the Drysdale River region. The black boxes indicate the location of EDS analysis suggesting the 'stem' of the stalagmite to be predominantly composed of quartz whilst the stalagmite 'head' displays well crystallised sulphate crystals.

oxalates. These minerals derive from a range of sources both within and outside the shelter which will be discussed in a separate paper. This study has allowed the suitability of each system to the application of multiple radiometric dating methods to be assessed. Accretions containing both uranium-bearing minerals such as phosphates and carbon-bearing minerals such as oxalates are especially promising in this respect. Understanding the occurrence and formation mechanisms associated with these mineral accretions is crucial for targeted sample collection and the application of a range of dating techniques as well as for the development of conservation strategies for this remarkable cultural legacy.

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