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Determining decay mechanisms on engraved rock art sites using pH, chloride ion and redox measurements with an assessment of the impact of cyclones, sea salt and nitrate ions on acidity

Ian D. MacLeod*
Western Australian Museum
Fremantle WA, Australia
ian.macleod@museum.wa.gov.au
Warren Fish
CBG Solutions
Kingsley WA, Australia
warren.fish@cbgsolutions.com.au
*Author for correspondence

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Abstract
This work reports on a series of physical, chemical and colour measurements conducted on petroglyphs under the direction of the traditional owners, the Murujuga Aboriginal Corporation, in the eponymously named national park located in the Pilbara region of Western Australia. A study of the way in which the surface pH, chloride ion activity and redox potentials respond to changes in the concentration of NO_x and NH_3 emitted from contiguous industries demonstrates that the decay mechanisms are controlled by pH-dependent mobilisation and precipitation of manganese minerals. The availability of water in this arid region controls the amount of acid produced through microbial metabolism. Decay mechanisms were discerned by plotting pH against redox potential. Localised irrigation of the rock surfaces provided quantitative assessment of the mobilisation of iron and manganese from the rock patina. Accidental release of ammonia, deposition of sea salts and periodic cyclonic rainfall all decreased acidity, which stabilises the engravings.

INTRODUCTION

The Dampier Archipelago is on the Indian Ocean coast of the Pilbara region of Western Australia and the Burrup Peninsula was created by bridging Dampier Island, the largest of the 42 islands and islets. The 118 km^2 of rugged landscape consists of boulder-strewn ridges and deep-sided valleys (Figure 1). The area is known as Murujuga, meaning ‘hip bone sticking out’ in the local Aboriginal language, and has an extremely high density of engraved rocks with more than 1,100 individual motifs per square kilometre (Bird and Hallam 2006). The petroglyphs were made by removing the outer deep red-brown weathered crust through percussion and scoring the rock surfaces to reveal the pale colours of the parent gabbro and granophyre rocks. The contrast diminishes with time leaving the oldest as faded images retaining the original relief. Dating by midden shells gives an age for the fully patinated engravings of 10,000–20,000 years, while the engraved thylacines (Tasmanian tigers) date to more than 3,000 years ago (Figure 2) when the animals became extinct on the mainland of Australia (Bird and Hallam 2006). More recently, Mulvaney (2011) has used sequential dating techniques to date the earliest engravings to around 40,000 years ago.

Figure 1. Typical steep-sided and boulder-strewn valley in the Murujuga National Park near site 4

Figure 2. Engraved image of 3,000-year-old thylacine in Happy Valley being assessed. Photo: Ken Mulvaney

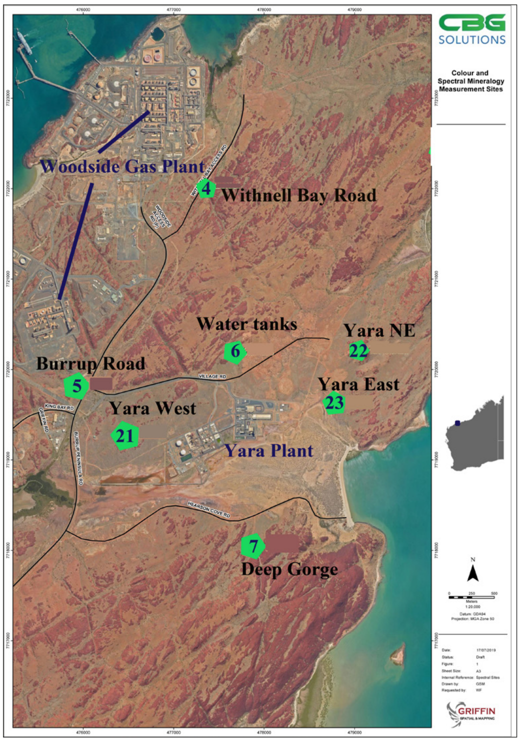

Figure 3. Partial map of Murujuga showing measurement points and Woodside and Yara industrial plants

In 2007, the Murujuga National Park was gazetted on Australia's National Heritage List in recognition of the area containing more than two million engraved petroglyphs. Aboriginal people believe that the images were created by their ancestors in the *Dreaming*. Murujuga hosts several large iron ore, liquefied natural gas, salt production and fertiliser facilities, all exported through nearby ports. Since the petroglyphs adjoin industrial areas, there has been strong public concern expressed that the rock art could be damaged by airborne emissions (FARA 2017). The perception is that the combined nitrogen oxides (NO_x) and sulfur oxides (SO_x) outputs would initiate acid dissolution of the rock patina and so diminish the contrast in the rock engravings, leading to the loss of heritage and tourism values.

During the development of the North West Shelf Venture gas production facility in the 1980s, Woodside Petroleum relocated hundreds of engraved rocks to a 'compound' on the leeward side of the adjacent hills. The responsibility for the engraved rocks was vested in the Western Australian Museum. Currently, Woodside exports over 4 million tons of liquified natural gas per annum (Figure 3). In 2002, the Western Australian Government established a rock art monitoring program in response to concerns about possible adverse impacts on the rock art from industrial emissions. The authors undertook the first in situ measurements on the 'compound' rocks and surrounding areas in 2003–2004 in response to concerns over management of aboriginal sites. This data indicated that as nitrate ion concentration increased above background levels, there was significantly increased microbial activity, which lowered the pH. The 'reactivity' of the rock surfaces was assessed through analysis of solutions collected from a portable 'dam', which had been filled with ultra-pure water. The solutions were expertly analysed in Perth for metal cations and anions, and the electrical conductivity provided a measure of the total soluble minerals. The data showed that there was measurable loss of iron, manganese and clay minerals from the most acidic surfaces (MacLeod 2005).

The government research also included measurements of air quality, microclimate, dust deposition, colour change, mineral spectrometry,

Figure 4. Site 7: gabbro rock with kangaroo motif at Deep Gorge

Figure 5. Site 5: granophyre rock with cormorant at Burrup Road West

Table 1. Locations of sites, distance from the NH_3 plant and orientation

Location	Site	Distance (km)	Direction
Withnell Bay Road	4	2.6	N
Burrup Road*	5	1.4	WNW
Water Tanks*	6	1.1	NNE
Deep Gorge*	7	1.4	SSE
Yara West	21	0.6	W
Yara North East	22	2.0	NE
Yara East	23	1.6	E

* Air quality monitoring stations adjacent to these sites

microbiological analyses, accelerated weathering studies, air dispersion modelling studies and detailed photographic recording. After seven years, the conclusion was that there was no measurable impact on the rate of decay of the rock art that could be assigned to the existing levels of pollutants. Assessment of the long-term impact of pollutants saw the mineralogy team of the Commonwealth Scientific and Industrial Research Organisation (CSIRO) use ammonia solutions at pH 11.5 and nitric acid solutions at pH 2 to simulate the impact of decades of exposure. The rock surfaces were examined by X-ray powder diffraction (XRD), scanning electron microscopy (SEM), Fourier transform infrared (FTIR) and spectroscopically, which showed that significant changes would only happen under conditions that would be dangerous to human health (Ramanaidou et al. 2017). Public health regulations would prevent such eventualities. The Friends of Australian Rock Art (FARA) rejected the findings, claiming that they were industry biased. From 2004 to 2015, the CSIRO teams also conducted annual spectrophotometric and colourimetric studies in the region. This work stopped in 2015 following trenchant criticism regarding methodology, which claimed that their experimental methods were both inaccurate and non-reproducible (Black and Diffey 2016).

Although this work was specifically designed to manage regulatory compliance for industry, the methodology is applicable to all rock art engraving sites.

TESTING METHODOLOGY

In 2017, Fish and MacLeod were contracted by the ammonia and ammonium nitrate producers Yara Pilbara Nitrates (Yara) to continue the CSIRO colour monitoring measurements on the six sites within a 2 km radius of the plants in the Burrup and to apply in situ pH, chloride, redox potential and photographic measurements to the chosen study areas. The sites selected by government inspectors included the coarse-grained gabbro rocks at Deep Gorge (Figure 4, site 7), Yara North East (Yara NE, site 22) and Yara East (Yara E, site 23). These gabbro sites were contrasted with the fine-grained granophyre rocks at Burrup Road (Figure 5, site 5), Water Tanks (site 6) and Yara West (Yara W, site 21) and at Withnell Bay Road (site 4), as shown in Figure 3 and Table 1. The latter site is 2.6 km from the Yara plant but is less than 300 metres from the continuously operating flare tower at the gas plant (Figure 3), which is only 150 metres south of the famous *Climbing Man* engraving, which has immense cultural significance to the local Aboriginal community. However, no images can be shared, as it is a male-only site.

The CSIRO method used spectrophotometric and colourimetric analyses of four spots at each site, for both background and engraved areas (Lau et al. 2008), that gave the differences in colour and mineralogy between 2017–2019. Because weathered rocks consist of oxides, hydroxy-oxides and clays, the mineralogical and colour changes are sensitive to the acid-alkaline balance of the surface and measurement of the surface pH was essential. Changes in the chloride concentration showed how much sea salt had been deposited on the rocks. Redox potentials indicated which metal ions are providing the energy available to microflora living on the rocks. Yara had established monitoring stations for collection of rainfall and air quality data on NO_x, SO_x and ammonia (NH_3) at Burrup Road, the Water Tanks and Deep Gorge (Figure 4). During the field measurement programs, the consultants undertook the training of young Aboriginal rangers.

EQUIPMENT

The pH data was recorded using a daily calibrated flat surface pH electrode (VWR model no. W7567287) connected to a TPS Aqua pH/ORP/°C meter. Surface pH readings were taken by instilling 1–2 drops of distilled water between the rock surface and the electrode. Stable values were obtained after 30–60 seconds. The calibrated (1,000 and 100 ppm) chloride ion activity was measured using a TPS WP-90 ion-pH-mV-°C meter coupled with an Orion Thermo 1609-186881 chloride ion-specific electrode. The rocks were wetted with two drops of a 0.05 M sodium nitrate solution to provide an electrolyte to stabilise the liquid junction between the sensing head and the rock surface. Stable readings of the chloride activity were obtained within a minute. Redox measurements were taken using a 2 mm o.d. platinum wire electrode pushed through a sponge rubber mat, wetted with local tap water, and an Ag/AgCl (3M KCl) reference electrode was held near the platinum electrode. The reference electrode was calibrated using a saturated quinhydrone solution at pH 4.0 and had a voltage of + 0.210 volts versus the standard hydrogen electrode. Colour measurements were made using the same Konica Minolta chromameter used by CSIRO (Lau et al. 2008).

GENERAL RESULTS: EFFECTS OF pH ON DISSOLUTION MECHANISMS

In the absence of pollutants, the natural pH of local weathered gabbro and granophyre is 5.5 ± 0.2, which arises from the hydrolysis of metal ions present in the surface patinas. Any significant deviation from this mean value will bring about changes in colour contrast; acidification leads to surface loss and alkalisation (from sea salt or ammonia leaks) causes fresh minerals to deposit and thereby change the appearance of the engravings. Each of the monitored rock surfaces can present different pH values year in and year out owing to localised microbiological populations (whose metabolites are acidic) (MacLeod et al. 1995), so it is not unusual to have standard deviations of the mean pH of ± 0.5 even on rocks as small as 30 × 30 cm (Figure 5). Thus, pH changes are only statistically significant if the differences in the means exceed the sum of the standard deviations associated with the set of 10–12 readings on each rock.

The Deep Gorge site was the only area that had a significant decrease in acidity, with the pH increasing from 5.7 pH in 2017 to 6.7 in 2018. Photographs showed possible subtle changes in the rock patination, with an increased amount of the purple-black patina on the right-hand side of the rock during the less acidic period. Analysis of the rainfall at the adjacent monitoring station showed that the decrease in acidity was due to significant leakage events from the NH_3 plant. However, there was no visually discernible effect on the rock art. During the following year, the background nitrate count increased from 0.4 to 2.1 ppm, which caused the mean pH at Deep Gorge to fall from 6.7 ± 0.4 to 4.4 ± 0.4 because of increased microbiological activity. The minimum pH of the rock was 3.3 and the maximum was 5.0 during the 2019 measurements. Without major rainfall from cyclones to wash the rocks, the rock acidity increased. Light showers and night-time dew formation provide enough moisture to keep the bacterial populations alive and to respond to the nitrate concentration. With only 9 ± 4 ppm of chloride at Deep Gorge it was not enough to provide sea salt buffering.

The significant pH changes at Deep Gorge between 2018 and 2019 provided the stimulus for detailed analysis, through plotting the pH and redox (voltage) measurements to discern the chemical equilibria controlling the dissolution and precipitation of minerals. During the 2018 measurements (^{mean} pH = 6.7 ± 0.4), it was noted that the reverse side of the large rock (where measurements were made) had a deep purple-black patina associated with the presence of Mn_3O_4 (Figure 4). The plotted data showed that the purple-black areas were consistent with the formation of Mn_3O_4 as shown in Equation 1:

$$3\,Mn^{2+} + 4\,H_2O \rightarrow Mn_3O_4 + 8\,H^+ + 2\,e^- \qquad (1)$$

The slope of the Pourbaix diagram for equation 1 was −4*0.0592 volts/pH or −237 mV/pH (Pourbaix 1974). This same E_h/pH response was found in 2019 at the cormorant image at the Burrup Road site (Figure 5) and at the Yara North East site, which shows it is a commonly observed precipitation process. The remaining 2018 points corresponded to the red-brown to black patination formation of manganese dioxide, which had a slope of −2*0.059 or approximately 118 mV/pH, as required by Equation 2:

$$Mn^{2+} + 2\,H_2O \rightarrow MnO_2 + 4\,H^+ + 2\,e^- \qquad (2)$$

However, the impact of the ammonia leak was lost by 2019 when the mean pH was 4.4 ± 0.4, so instead of forming insoluble manganese minerals, the patina was dissolving, according to Equation 3. Soluble manganous ions (Mn^{2+}) were being oxidised to the dihydroxy Mn^{4+} ion, $Mn(OH)_2^{2+}$, with a slope of −29 mV per pH, which reflects the 1:2 ratio of protons to electrons shown in Equation 3:

$$Mn(OH)^+ + H_2O \rightarrow Mn(OH)_2^{2+} + H^+ + 2e^- \qquad (3)$$

The ability to pick up such changes through the use of pH and redox measurements demonstrates that such in situ measurements form an indispensable tool in monitoring rock art sites and in determining the decay mechanisms.

This sensitivity to subtle changes in pH, altering the colour contrast of the measurement points at Deep Gorge, explains why the CSIRO team reported colour differences in one year that sometimes disappeared in the subsequent year (Lau et al. 2008). The changes in the contrast occur as a result of dissolution of old minerals and precipitation of new surface minerals. This variability in the colour and appearance of engraved rocks had been perceived by Black et al. (2017) as a sign of poor experimental work, but our work showed otherwise.

The pH data in Table 2 shows that for the gabbro rock at the Yara East site (no. 23 in Figure 3) there was a steady decline in pH between 2017 and 2019 and the patina subtly changed. The minimum pH in 2018 was 4.8, which explains why Equation 2 (oxidation of Mn^{2+} to insoluble MnO_2) was the preferred mechanism, while the more acidic minimum of 4.5 in 2019 saw oxidation of Mn^{2+} to $Mn(OH)^{2+}$ (Equation 3) or the loss of manganese from the patina.

Table 2. Mean rock surface pH for 2017–2019 at monitoring points

Site no.	Location	2017 pH	2018 pH	2019 pH
23	Yara East	5.5	4.8	4.5
22	Yara North East	6.2	6.0	4.6
21	Yara West	6.4	5.8	5.2
7	Deep Gorge	5.6	6.7	4.4
6	Water Tanks	5.7	6.0	4.7
5	Burrup Road	5.2	5.0	4.8
4	North Withnell Bay Road	3.8	5.5	5.1
4a	North Withnell Bay Road (10 metres north)	3.9	4.3	4.4

IMPACT OF RAIN EVENTS AND SEA SPRAY

The Murujuga environment is arid and hot with a low annual rainfall of 260 mm and an evaporation rate of at least 3,200 mm. Mean winter temperatures are 20 ± 2°C and the mean summer temperature is 32 ± 3°C, with the rock surfaces varying between 43°C–57°C. These desert conditions tend to inhibit biological activity. Previous work has shown that within a day of a rainfall event, there is a measurable increase in microflora activity (MacLeod 2005). Between the initial pH measurements in the Murujuga National Park in 2003–2004 there were six cyclonic rainfalls when an average of 174 ± 50 mm of rain fell within 24 hours. Such events drench the rock surfaces and 'reset' the acid clock. A rainfall event took place on 6 June 2018, between the 2017 and 2018 measurements, which 'reset' all the sites, other than at Deep Gorge, which was impacted by the ammonia leak (Table 2). With no major rainfall between 2018 and 2019, the normal pattern of increasing acidification on the sites occurred. It was noted that there are no systematic pH differences between the gabbro and the granophyre rocks (Table 2). The changes at the Withnell Bay Road site and its surrogate rock indicate that a highly localised wetting event occurred immediately prior to the 2019 measurements. On 7 February 2020, a cyclone dumped 234 mm of rain on the region, so a monitoring visit will take place in September 2020 to confirm that this has 'reset' the pH.

The changes in the mean pH on the monitoring sites between one year and the next, or between consecutive years, is directly dependent on the change in chloride concentration, as shown by Equation 4. The dried sea salt provides buffering for the rocks, since acidity from microbiological activity is neutralised with increasing salt content:

$$\Delta^{mean}\,pH = -\,0.27 + 0.0037\,\delta[Cl]_{ppm} \tag{4}$$

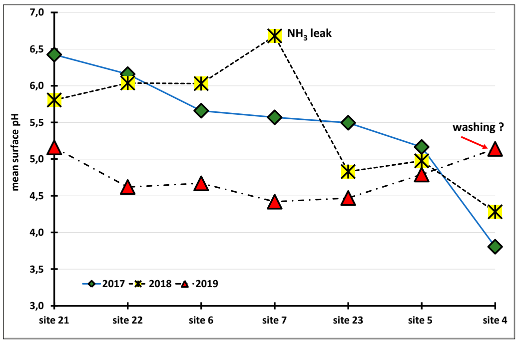

Figure 6. Plot of mean pH for the monitoring sites between 2017–2019 showing seasonal variations, including ammonia release events and localised washing event at site 4 in 2019

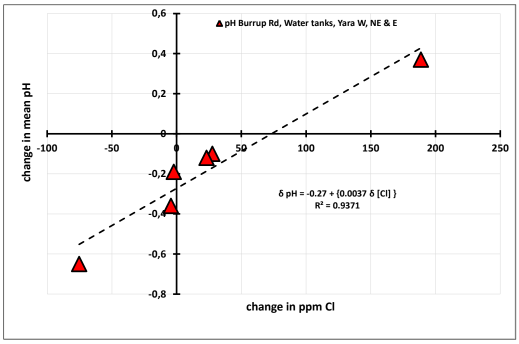

Figure 7. Plot of ΔpH between yearly median data vs. the change in seasonal salt levels (δ[Cl]) for 2019 compared with 2018 and 2017

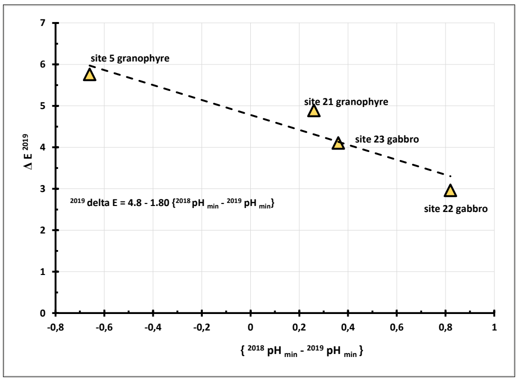

Figure 8. Plot of ${}^{median}\Delta E_{2019}$ for colour contrast between background and engraved areas decreasing with increasing alkaline shift in the minimum pH between 2018 and 2019

In this equation, the change in sets of pH measurements is noted as Δ^{mean} pH and the increases or decreases in chloride activity as $\delta[Cl]_{ppm}$. There was a very high degree of correlation between the change in pH and the change in chlorinity, as shown by the high R^2 of 0.937 with a 15% error for both the intercept and the slope. If the chloride concentration decreases between inspections (rain), the sites become more acidic and vice versa (Figure 7). The non-zero intercept value in Equation 4 is due to acidity associated with nitrate concentration.

EFFECTS OF NITRATE ON pH

When the monitoring began in August 2003, the mean NO_3^- was 6.3 ± 5.1 ppm with a maximum of 19 ppm. Six months later, the mean had fallen to 4.5 ± 3.7 ppm with a maximum of 9 ppm. Coincident with our reports on background NO_3^-, Woodside Petroleum introduced low NO_x burners between 2005 and 2015, which resulted in greatly reduced nitrate levels of 0.7 ± 0.5 ppm during 2017–2019 monitoring. The overall acidity of the rocks is now lower than in 2003–2004. The direct correlation between the median pH and the nitrate concentration on the rocks was found for the Withnell Bay Road, Burrup Road, Water Tanks and Yara West sites, as seen in Equation 5, where the pH fell by 0.9 per ppm nitrate:

$${}^{2019}pH_{median} = 5.5 - 0.9\ (\pm 0.3)\ [NO_3]_{ppm} \qquad (5)$$

The intercept value of 5.5 ± 0.3 is the natural pH of local rocks. The error in the pH slope reflects the R^2 of 0.72 due to the variability of pH associated with the surface salt (see Equation 4).

EFFECT OF MINIMUM pH ON COLOUR DIFFERENCE

All the colour measurements were made using the CSIRO chromameter. Twenty repeat measurements on each background and engraving point (four locations) were made to improve reliability, but at times this was counterproductive. When the median ΔE values for the change in colour difference at Burrup Road (site 5), Yara West (site 21), Yara North East (site 22) and Yara East (site 23) were plotted against the change in the minimum pH (2018 compared with 2019), the ΔE values decreased (the contrast becomes less) as the pH difference became more alkaline, as shown in Equation 6 and Figure 8:

$${}^{background.\,-\,engraving}\Delta E_{2019} = 4.8 - 1.8\ \delta\, pH_{min} \qquad (6)$$

The variability in the colour measurements was high and data from Deep Gorge did not fit the regression owing to the exposure to ammonia vapours in 2018. The colour contrast at the Water Tanks was statistically unreliable owing to the very weathered nature of the engravings. These changes are consistent with the outlined mechanisms in which more manganese minerals are precipitated as pH increases, which serves to diminish colour contrast. Owing to the nature of the measurements, the R^2 for Equation 6 was relatively low at 0.88, which gave an intercept error of 6% and a 25% error for the slope. Without pH monitoring data, it is unwise to base conservation management decisions solely on colour differences between the engraved and background areas of the images.

CONCLUSION

Analysis of the in situ pH, chloride and redox data has resulted in the first definitive answers to the factors controlling the decay mechanisms on engraved rocks in the Pilbara region of Western Australia, in which mobilisation and precipitation of manganese minerals are the dominant reactions. From the way in which the pH and the voltage of the electrochemically active iron and manganese minerals changed, it was possible to construct thermodynamic stability diagrams and determine the active species. Subtle changes in surface pH of only 0.3 saw the surface chemistry of the rocks change from dissolution of manganese minerals, resulting in increased colour contrast, to precipitation of dark oxidised manganese minerals, which decreased the colour contrasts between the engraved and background areas of the petroglyphs. A combination of cyclonic rainfall events, dry deposition of ammonia and wind-borne sea salts works to mitigate the release of iron and manganese minerals from the Murujuga rock engravings. Comparison of the early surface washing data collected in 2003–2004 and that collected between 2017 and 2019 showed that the reduced amounts of soluble nitrate are responsible for stabilising the iron-rich minerals in the patinas with a concomitant reduction in surface acidity.

The microclimate of the individual rock surfaces and the activity of the microflora control whether the colour differences between engraved and background rock are enhanced or diminished. The present monitoring data shows that there is presently no adverse impact on the rock engravings from industrial pollution owing to a lower NO_x level than when the studies commenced 14 years ago. It is strongly recommended that future research on all petroglyph sites incorporates the methodology associated with this work, since the combination of pH and redox data is essential for determining the decay mechanisms. For other rock art sites away from the influence of sea salt spray, the chloride measurements could possibly be dispensed with. This research program has become integral to the development of skills in the local youth who form the bulk of the indigenous Ranger Program run by the Murujuga Aboriginal Corporation.

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