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Title: Provenance tracing using mother-of-pearl and the impact of button processing on the material's trace elemental and isotopic concentrations

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Garment buttons and other artefacts incorporating mother-of-pearl (nacre) are significant items of material culture frequently found on archaeological sites and in museum collections. From around 1860 until the end of the Second World War, Australia was the foremost international source of mother-of-pearl for buttons, which were mechanically mass produced during that time. Since nacre formation is impacted by climate, water chemistry and other environmental factors at the time of shell formation, mother-of-pearl is potentially well suited to geochemical investigations of provenance. And yet, this avenue of inquiry, and the impact of button manufacturing on the compositional nature of mother-of-pearl, have received little attention to date. This study comprised stable carbon ($\delta^{13}\text{C}$) and oxygen ($\delta^{18}\text{O}$) isotope analysis of six mother-of-pearl samples from at least four different species sourced from the major pearling areas of Australia. It found oxygen isotope to be an effective marker in discriminating between most sample source locations, although some regions were not distinguishable. Additional and much higher density sampling and analysis of modern pearl shell might resolve this issue. The study also determined that mechanical processing during button manufacture has no significant impact on the trace element and isotope compositions of the mother-of-pearl. In doing so, it demonstrates the potential for source tracing mother-of-pearl material culture items to shed new light on the history of the pearling industry in Australia, one of the country's most historically important maritime industries.

Introduction

Pearling is an historically important marine industry for Australia, which was the foremost international source of nacre, more popularly known as pearl shell or mother-of-pearl, for button manufacture from 1860 until the end of the Second World War. Consequently, artefacts incorporating MOP are significant items of material culture frequently associated with archaeological sites and museum collections. Mother-of-pearl is a composite material composed of interlaid layers of aragonite (a crystalline form of calcium carbonate) and biopolymers, and so is amenable to geochemical investigations of palaeoclimate, provenance, diet and water chemistry. In spite of this, few studies have investigated the effect of button processing on the trace element and isotopic composition of this material. Determining the possible presence and extent of elemental and isotopic leakage or uptake during the manufacturing process is critical, as it could significantly impinge on the ability of geochemical methods to characterise mother-of-pearl effectively, with significant implications for determining the provenance of archaeological objects fabricated with this material.

The present research trials the use of oxygen and carbon isotope analysis using isotope ratio mass spectrometry (IRMS) and trace elemental analysis using laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) on mother-of-pearl shell pairs from a variety of species from noteworthy pearling locations in Australia. For each shell sample, one half of the shell was manufactured into a button using traditional manufacturing methods, and then both shell halves (processed and unprocessed) were analysed to determine if the button manufacturing process had any effect on the elemental and isotopic matrix of the sample. The results show that the isotope and trace element compositions of mother-of-pearl are heterogeneous on both an intra- and inter-sample basis. This is interpreted to be the result of environmental conditions, namely variations in water chemistry, temperature, and nutrient availability during the growth of the shells. There also appears to be no systematic effect of button processing on the geochemistry of pearl shell. Further analysis of mother-of-pearl grown under controlled conditions is proposed to investigate these parameters in more detail.

Historical background to the study

Pearls became an important luxury item from about 7,500 BP (Carter 2005; Charpentier et al. 2012:1). Pearls are used principally as a decorative item in jewellery and clothing, although occasionally they are crushed and added as an ingredient to cosmetics and medicines. Pearl oyster shell, specifically the inner layer of nacre and henceforth referred to as mother-of-pearl, is best known as a material manufactured into garment buttons (Yamaguchi 1993:497), but was used in a range of objects, including gaming tokens, cutlery, hair combs and jewellery items, as well as to make objects of art and as inlay for furniture.

Figure 1. Wet cutting lathe used to cut button blanks from shell (G. Hook, 2016).

Indigenous Australians were using and trading pearls and mother-of-pearl for personal adornment long before European contact (Balint 2012:546; Berndt and Berndt 1979:247; McCarthy 1994:76–77). William Dampier in 1699 noted the presence of oysters in the Shark Bay area of Western Australia and recognised the commercial potential for pearling (Bach 1955:4; Edmonds 1996:4). The first commercial pearling licence for Shark Bay was granted in 1851, but the industry only became well established in the latter half of the 1860s, and then mainly in the Nickol Bay area of the Pilbara and the Torres Strait (Anderson and Green 2011:6; Bach 1955:4–5; McPhee 2001:2;). The pearling industry in Australia eventually expanded to reach from the Pilbara to the northern tip of the Great Barrier Reef (Anderson and Green 2011:6–7; McPhee 2001:1), forming one of the largest pearl fields in the world (Balint 2012:546; McPhee 2001:1). By the turn of the 20th century, Broome was the principal port of trade for pearls and mother-of-pearl (Mullins 2005:215). Currently, Australia is the leading international exporter of pearls, with the industry's main centre located in Broome.

Mother-of-pearl has been used extensively in the manufacture of buttons since the early 1800s, with the best-known historical example of a mother-of-pearl button coming from King George IV's undershirt dating to 1827 (Lindbergh 1999:51). Nevertheless, mother-of-pearl buttons from archaeological contexts predate this. For example, the excavation of *Rapid*, an America-China trader that wrecked in 1811 at Ningaloo reef, Western Australia, yielded mother-of-pearl buttons (WA Museum, reg. nos RP5050 and 5389).

As pearling gained popularity in Australia during the mid-19th century, mother-of-pearl exports increased, as did pearl oyster mortality, following their transportation from the pearl shell fields to processing sites onshore (Pass et al. 1987:149). As a direct result of this influx of mother-of-pearl, demand increased as well and the Australian large silver lipped oyster (*Pinctada maxima*) accounted for 80–90% of the mother-of-pearl made into buttons worldwide, with all trade being conducted in London (Mullins 2005:217).

Mother-of-pearl button manufacturing

Mother-of-pearl was used extensively for the manufacture of buttons before being replaced by inexpensive Bakelite, plastics and zippers during the World Wars. Mother-of-pearl buttons were originally hand-made, like those of bone, but became mechanically mass-produced from the mid-19th century on (Lindbergh 1999:51).

The mechanical production of mother-of-pearl buttons involves several stages. The first step in button manufacture is the cutting of the button blank by a hardened-steel or diamond-tipped core drill (Figure 1). This can be done using the dry cut method or with water to cool the mother-of-pearl, depending on manufacturer choice and the available machinery. Buttons may be soaked for a week in water prior to blank cutting when using the dry cut method (Claassen 1994:53; Marcel 1994:7). The cut button blanks are ground down to the required thickness on a 60-grit carborundum-grinding wheel (Figure 2), and one side is flattened to serve as the back of the button. Next, the blanks are 'backed' to expose more of the nacre and remove the rough exterior of the shell, known as bark (Perfect 2001:114). Blanks also may be tumbled in pumice and/or water to remove the bark and to aid in rounding the edges (Claassen 1994:55). The ground blanks can be left plain, if desired, or turned on a small lathe to add decorative patterns (Figure 3; Perfect 2001:115).

Buttonholes (usually two or four in total) are added using a small drilling machine (Figure 4). This machine will only drill one hole at a time, but the blank can be rotated without being taken

out of the machine (Claassen 1994:55). At this stage, buttons also may be dyed or bleached (Marcel 1994:8).

The final step in the button manufacturing process is polishing the button. This is undertaken either by hand, using a polishing mop and a polishing compound, or by ‘barrelling’ in a tumble barrel. In either case, a minute amount of hydrochloric acid is used in the first round of polishing, followed by rinsing with cold water. The final polish is done with a small amount of sulphuric acid diluted with cold water. When barrelling, steam, sawdust and washing powder occasionally are used as well to aid in the polishing (Claassen 1994:55–56).

Figure 2. Carborundum grinding wheel (60-grit) used to grind down mother-of-pearl blanks to desired thickness and remove bark from button exteriors (G. Hook, 2016).

Figure 3. Turning lathe used to add patterns to buttons (G. Hook, 2016).

Figure 4. Small drill used to bore buttonholes (G. Hook, 2016).

Geochemistry of mother-of-pearl

Mother-of-pearl is formed of layers of hexagonal plates of aragonite interlaid with sheets of organic biopolymers. This shiny, multi-coloured material, which forms the interior of many mollusc shells, is extremely strong and fracture resistant (Wang et al. 2001). The isotope and trace element compositions of mollusc shells can record important information about the physiological and environmental conditions at the time of shell formation (Gifford et al. 2004:28; McConnaghey and Gillikin 2008:287). Compared to other bivalves, pearl oysters pump the largest volume of water through their systems and so are particularly sensitive to their environment and contaminants (Gifford et al. 2004:28).

Many variables may change the isotopic and trace element composition of a pearl shell. The elemental composition can be influenced by the size and sex of the mollusc, season, salinity, water temperature, seasonal contaminant interactions in the water column and localised pollution (Gifford et al. 2004:29). The carbon isotopic composition in marine molluscs is complex, but is controlled largely by salinity, nutrient availability, the local CO₂/O₂ ratio and the availability of dissolved inorganic carbon (McConnaghey and Gillikin 2008). Water temperature and salinity principally control the oxygen isotope composition of marine molluscs (Eerkens et al. 2005:1504; Eerkens et al. 2007:181; Eerkens et al. 2010:107; Keith et al. 1963:1783). Post-depositional factors also can change the trace element and isotopic compositions of the shell matrix, particularly when pearl shell are buried within an archaeological site (Cox 1996:52; Eerkens et al. 2007:176, 188), but this is beyond the scope of the present study.

Figure 5. Australian source locales for pearl shell samples (V. Sullivan, 2016; Landsat image from Google™ earth, data from SIO, NOAA, US Navy, NGA GEBOC).

Despite the sensitivity of marine molluscs as recorders of environmental conditions, mother-of-pearl buttons have not previously been the focus of geochemical investigation of provenance. Instead, mother-of-pearl buttons usually are studied in terms of typology (Claassen 1994; Garrett 1990; Lindenbergh 1999; Marcel 1994). In most archaeological literature, the mother-of-pearl for buttons is identified anecdotally as coming from Australian waters (Mienis 2003:8; Thurston 2004:94), although shell also was harvested in the Philippines, Fiji, Singapore, Japan and Indonesia (Bach 1955:180; Cox 1996:56; Martinez 2012:231; Mienis 2003:8; Yamaguchi 1993:498). The present study aims to overcome such anecdotal references by analysing oxygen, carbon and trace element compositions of mother-of-pearl buttons to determine if IRMS and LA-ICP-MS can be used to provenance manufactured mother-of-pearl buttons.

Methods

This study comprised the analysis of six motherof-pearl samples. These samples were modern shell sourced from the major pearling areas of Australia (Figure 5) and included a range of species (Table 1).

Table 1. Source Location and Species of Samples Analysed

Sample	Collection location	Shell species
1	Broome	<i>Pinctada maxima</i>
2	Darnley Island	<i>P. maxima</i>
3	Houtman Abrolhos	<i>P. fucata</i>
4	Houtman Abrolhos	<i>P. margaritifera</i>
5	Houtman Abrolhos	<i>Ptera penguin</i>
6	Darwin Harbour	unknown

Geochemical analysis was performed on material taken from complete shells (both halves), with one half cut into buttons and the other half left unmodified. As much as possible, the samples for analysis were taken from the same region on each shell of the matched pairs in order to minimise the impact of geochemical variations between growth bands. Unprocessed samples were taken from the shell using a titanium spade drill bit and file and any bark adhering

to the nacre was removed using pliers. The sampling equipment was cleaned after each shell was sampled.

Figure 6. Mother-of-pearl buttons produced by George Hook (C. Jordan, 2016).

George Hook, proprietor of George Hook & Co. of Birmingham, UK, fabricated all of the buttons used in this study. He produced both plain and edged buttons using the traditional manufacturing process described above (Figure 6), and hand polished each one. After manufacture, each was halved to facilitate analysis. Hook did not make any buttons from Darwin shell (shell 6), as he considered it “dead shell” that would simply have crumbled into fragments during the manufacturing process.

Samples for stable carbon and oxygen isotope analysis ($\delta^{13}\text{C}$ and $\delta^{18}\text{O}$, respectively) were crushed manually and then powdered using a micro ball-mill equipped with stainless-steel chambers. Samples of carbonate powder were flushed with ultra-high purity helium (99.999%) in 12-ml vials and reacted with 0.05 ml of 100% phosphoric acid at 50°C for one hour (Paul and Skrzypek 2006:2035; Paul and Skrzypek 2007:182). The stable isotope compositions were analysed at the University of Western Australia using a Thermo-Fisher Delta XL Isotope Ratio Mass Spectrometer coupled with GasBench II online gas preparation and introduction system. All results are expressed using the standard δ notation ($\delta^{13}\text{C}$ and $\delta^{18}\text{O}$) and are reported in per mil after normalisation to the Vienna Pee Dee Belemnite isotope scale (‰ VPDB). The multi-point normalisation was based on three international standards: NBS18, NBS19, and L-SVEC, each replicated twice (Skrzypek 2013:2817). The analytical uncertainty (1σ) was $\pm 0.10\text{‰}$ for $\delta^{13}\text{C}$ and $\pm 0.15\text{‰}$ for $\delta^{18}\text{O}$.

LA-ICP-MS analyses of buttons and raw shells were conducted at Southern Cross University using an Agilent 7700 Series ICP-MS coupled to a NWR213 (Nd:YAG deep UV [213 nm]) laser ablation system. Each sample was laser ablated using a scanning speed of 5 $\mu\text{m}/\text{sec}$ and repetition rate of 10 Hz in a continuous track measuring 60 μm wide and 10 μm deep. Robust signal intensities were obtained for magnesium (Mg), chlorine (Cl), potassium (K), calcium (Ca), titanium (Ti), iron (Fe) and strontium (Sr). To ensure accuracy and reproducibility of results, the NIST 613 international standard was measured at the start and end of each run.

Results

All geochemical results are presented with prefix S for unprocessed shell and B for button, with the geographic location corresponding to the data shown in Table 1. The results from the ^{13}C and ^{18}O isotope analyses are shown in Table 2. $\delta^{13}\text{C}$ values range from 0.36 to 2.62‰ VPDB, and $\delta^{18}\text{O}$ values range from -1.38 to 0.50‰ VPDB. The concentrations obtained for a selection of measured elements are presented in Table 3. Calcium concentrations range from 1.6 to 20.3‰, magnesium concentrations from 0.8 to 127.6‰ and strontium concentrations from 21.4 to 38.0‰. Trace metal concentrations are generally low, although some samples (S4 and S5) have elevated titanium and iron concentrations, while S6 has an elevated iron concentration.

Table 2. Oxygen and Carbon Isotope Results for Buttons (B) and Shell (S)

Sample	$\delta^{13}\text{C}$ (‰ VPDB)	$\delta^{18}\text{O}$ (‰ VPDB)
B1	2.62	0.50
B2	1.59	-1.32
B3	0.36	0.32
B4	1.14	0.36
B5	0.81	-0.15
S1	2.31	0.36
S2	1.51	-1.38
S3	0.28	0.29
S4	1.11	0.31
S5	0.91	-0.12
S6	1.03	-0.86

Table 3. Results of LA-ICP-MS Analysis of Shell Samples and Buttons (‰)

Sample [*]	Mg	Cl	K	Ca	Ti	Fe	Sr
B1	2.243	3.230	1.852	8,968	0.054	0.085	28.00
B2	3.162	1.631	1.190	9,712	0.250	0.073	25.26
B3	2.250	3.698	2.639	8,804	0.094	0.060	24.74
B4	1.756	2.135	1.051	9,889	0.189	0.060	25.65
B5	0.844	1.972	1.248	8,536	0.118	0.042	21.39
S1	1.760	3.911	1.653	8,391	0.411	0.089	23.28
S3	127.6	20.32	5.122	9,380	0.136	0.139	21.91
S4	26.06	5.458	2.778	14,113	289.4	1.554	23.42
S5	4.593	3.302	1.511	9,468	5.151	0.277	24.30
S6	5.367	3.166	1.532	11,107	0.233	1.169	37.96

^{*} Sample S2 was not analysed due to size restrictions.

Discussion

The results of oxygen and carbon isotope analyses clearly demonstrate that the manufacturing process has no significant impact on the composition of mother-of-pearl buttons. In particular, the $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ values of the processed and unprocessed samples are, in most cases, statistically indistinguishable from each other (Table 4).

In contrast, the four broad geographic regions (Darnley Island, Darwin, the Abrolhos Islands and Broome) from which the pearl shell was sourced are clearly differentiated from one another by the oxygen isotope value ($\delta^{18}\text{O}$) of the shell, with the exception of sample 5 (discussed below). If theoretical seawater values are calculated based on the $\delta^{18}\text{O}$ results from buttons and unprocessed samples using the equation of Grossman and Ku (1986) and a hypothetical $\delta^{18}\text{O}$ seawater value of -0.28‰ (Shackleton and Kennett 1975), then the results show the extent of differentiation between sample locations. This determination breaks down when comparing regions with different salinities, as demonstrated by the anomalously low calculated temperature at Darnley Island, which is attributed to the discharge of fresh water from the Fly River. Nevertheless, it is a useful comparative tool within regions.

Sample 5 from the Abrolhos Islands has an oxygen isotope value that is distinct from samples 3 and 4 from the same region. Corals from the Abrolhos Islands have a wide range of $\delta^{18}\text{O}$ values, which is believed to result from the sudden changes in water temperature driven by variations in the Llewelyn Current (Kuhner et al. 1999).

Sample 2 from Darnley Island shows significantly lower $\delta^{18}\text{O}$ values than all other samples. This is interpreted to be the result of freshwater discharge from the Fly River, which has been observed to affect the barium/ calcium ratios of corals in this region (Shen 1993). The $\delta^{13}\text{C}$ values for sample 3 differ significantly from those for samples 4 and 5, despite all samples coming from the Abrolhos Island region. This echoes the $\delta^{13}\text{C}$ results of analyses of coral samples from the region by Squire et al. (2013:108), which range from -1.9 to 0.3‰ . The source of these low values for corals is not well explained, but the degree of variability displayed in both studies suggests that $\delta^{13}\text{C}$ is not a viable tool for provenancing marine carbonates in this region.

Table 4. Calculated Water Temperature Based on Average Seawater Oxygen Isotope Composition

Sample	$\delta^{18}\text{O}^*$ (‰ VPDB)	Calc. temperature (°C)
B1	0.50	21.79
B2	-1.32	17.45
B3	0.32	21.36
B4	0.36	21.46
B5	-0.15	20.24
S1	0.36	21.46
S2	-1.38	17.31
S3	0.29	21.29
S4	0.31	21.34
S5	-0.12	20.31
S6	-0.86	18.55

* External error (1σ) for $\delta^{18}\text{O} < 0.15\text{‰}$.

Sample 6 from Darwin Harbour shows elevated iron levels, which is consistent with the reported high levels of heavy metals in sewage discharged in this area (Padovan et al. 2012:275). Trace element results from all samples show greater variations between the buttons and unprocessed shell in titanium and in trace element ratios related to environmental conditions, such as strontium/ calcium and magnesium/calcium (Table 3). This is surprising, given the widespread use of these proxies in the marine mollusc and coral literature. In the absence of further experimental data, this is attributed to the use of NIST 613 during laser ablation analysis, rather than a matrix-matched carbonate standard. No systematic change due to button manufacturing is evident in the other trace element ratios, so we do not attribute these unusual results to such processing.

Trace element values are reported in this paper as mean values for the shell; however, LA-ICP-MS provides the opportunity for spatially resolved analysis at much higher resolution. This provides the opportunity to explore intra-sample variations, such as caused by seasonal changes. The results are not reported in this fashion, as these high-resolution changes generally

are insignificant for provenance studies. Nevertheless, this approach could be useful potentially for examining sample contamination or seasonal variations. Overall, the most exciting aspect of this study is the ability of $\delta^{18}\text{O}$ to distinguish between biogenic carbonates from different regions. This result should be expected, based on regional maps of seawater $\delta^{18}\text{O}$ values (e.g., Schmidt et al. 1999); however, the potential of this approach has not been tested previously in a maritime archaeological context in Australia. Our results suggest a productive avenue of inquiry for provenancing mother-of-pearl material culture items to understand more about the history of the Australian pearling industry. conclusion

This study explored the potential for provenancing mother-of-pearl using trace element and stable isotope methods in order to facilitate new investigations of pearl shell trade using material from museum collections and archaeological sites. In particular, it investigated the effect of button processing on the composition of mother-of-pearl. This study found no evidence that the button manufacturing process affected oxygen or carbon isotope values or trace element levels in any way.

Oxygen isotope was an effective tool in discriminating between some sample collecting locations, although some regions (Houtman Abrolhos and Broome) were not distinguishable. Nonetheless, the lack of effect from button processing suggests that this area of research has significant potential; further and much higher density sampling and analysis of modern mother-of-pearl shell might resolve these issues. This would provide a new analytical tool to help understand one of Australia's most historically important maritime industries.

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Notes

1 $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ are measures of the ratio of stable carbon ($^{13}\text{C}/^{12}\text{C}$) and oxygen ($^{18}\text{O}/^{16}\text{O}$) isotopes, respectively. Stable isotopes are natural tracers, the analysis of which can serve as source indicators and be a useful tool for understanding complex ecological—in this case, marine—systems and processes (Fry and Sherr 1984:13–14; on the use of stable isotopes in archaeology and more broadly, see additional bibliography therein and also Ehleringer and Rundel 1989).

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