Radiocarbon Age Dating of 1,000-Year-Old Pearls from the Cirebon Shipwreck (Java, Indonesia)

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The 10th-century Cirebon shipwreck was discovered in 2003 in Indonesian waters. The excavation yielded an incredible array of archaeological finds, which included pearls and jewellery. Radiocarbon dating of the pearls agrees with the age of the shipwreck, which previously was inferred using recovered coins and ceramics. As such, these are some of the oldest pearls ever to be discovered. Based on this example, the present article shows how radiocarbon age dating can be adapted to the testing of historic pearls. The authors have further developed their sampling method so that radiocarbon age dating can be considered quasi-non-destructive, which is particularly important for future studies on pearls (and other biogenic gem materials) of significance to archaeology and cultural heritage.

The Journal of Gemmology, **35**(8), 2017, pp. 728–736, http://dx.doi.org/10.15506/JoG.2017.35.8.728

Introduction

The discovery of the Cirebon (or Nan-Han) shipwreck in the Java Sea in 2003 marks one of the most important archaeological finds in Southeast Asia in recent years (Hall, 2010; Liebner, 2014; Stargardt, 2014). Apart from ceramics, glassware and Chinese coins dating from the 10th century AD, the excavation of this ancient merchant vessel also produced a number of carved gastropod shells (presumably ritual objects, from *Turbinella pyrum*), jewellery (e.g. earrings with diamonds and sapphires), loose gemstones (e.g. sapphires, red garnet beads and rock crystal carvings) and a rather large number of small pearls (Tan, 2007; Liebner, 2010, 2014; Henricus, 2014). Of the more than 12,000 pearls that were recovered, most were less than a few millimetres in diameter (e.g. Figure 1).

Fishermen discovered the wreckage site accidentally in 2003, at depths greater than 50 m (Liebner, 2010) off the northern coast of Java, Indonesia, near the city of Cirebon (Figure 2). Excavation efforts were complicated due to legal uncertainties as to which companies/entities should be permitted to excavate the site, unfortunately leading to a period in which looting of the wreck occurred. Administrative, legal and diplomatic problems pertaining to the excavation, storage and ownership of recovered items continued in the following years (Tjoa-Bonatz, 2016).

The exact route of the ship is still disputed in academic circles (Liebner, 2014), but there is ample evidence of strong trading ties between China and western Asia, which are supported by shipping routes along the Strait of Malacca between the Malay Peninsula and the Indonesian

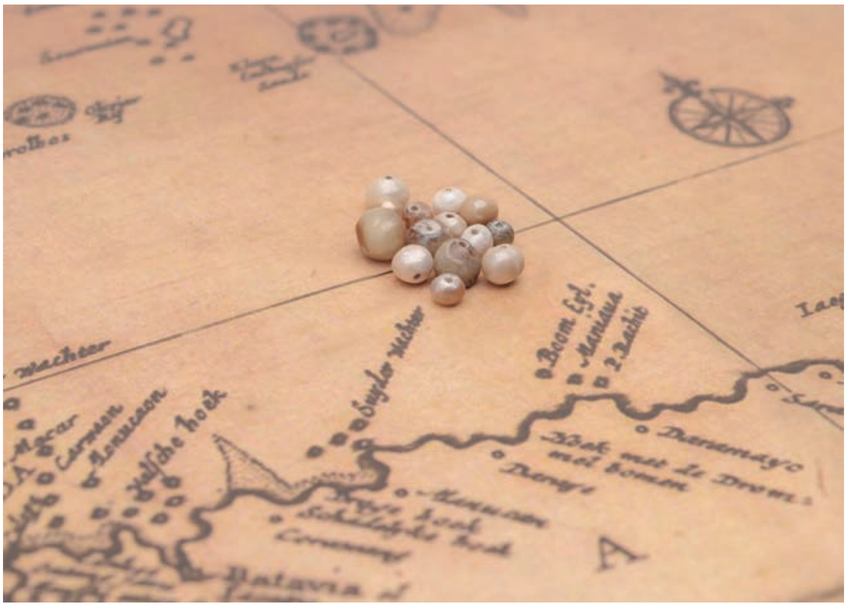
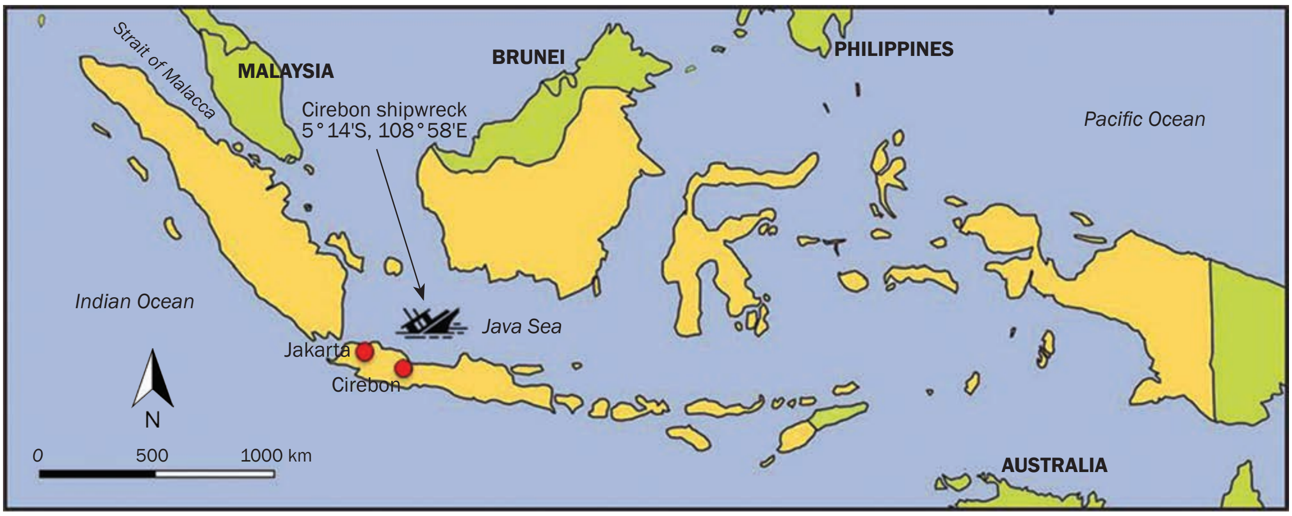


Figure 1: A small selection of pearls (approximately 2–8 mm diameter) from the Cirebon shipwreck was investigated for this study. The pearls are shown on a historic map of the Java Sea, where the shipwreck was discovered. Photo by Luc Phan, SSEF.

island of Sumatra in the 8th–10th century AD (Stargardt, 2014; Manguin, 2017; Shen, 2017). The recovery and study of artefacts from the Cirebon shipwreck offer a rare glimpse into trading practices of that period. It is thought that the trade in Yue ceramics (Chinese stoneware) peaked in the 10th century (Liebner, 2010), and they were a major export commodity during the Tang dynasty (Flecker, 2000). The form and decorations (including motifs) on Yue ceramics recovered from the wreck suggest a 10th-century period of manufacture and are complemented by potters' marks indicating 968 AD (Liebner, 2014). Furthermore, of nearly 5,000 individual coins recovered from the Cirebon shipwreck, eight were identified as "*Zhou Yuan tong bao*, a 955/6 issue by Shizong, emperor of the Later Zhou, mainly fashioned from confiscated 'Buddhist statuary of bronze [that] was mandated for recasting as coin' (Ouyang 2004: 115)" (Liebner, 2014, p. 197). Therefore, the coins and other recovered artefacts provided good evidence for a 10th-century age of the shipwreck. This time period corresponds to an era of upheaval in China called the 'Five Dynasties and Ten Kingdoms' during approximately 907–960 AD (Lorge, 2011); it was preceded by the Tang dynasty and succeeded by the Song dynasty (960–1127 AD). The pearls dated in this article yield further evidence documenting the 10th-century age of the shipwreck, and this provides an opportunity to better understand the rich history of this period in time.

Figure 2: The 10th-century Cirebon shipwreck is situated in the Java Sea, north of the city of Cirebon on the island of Java. The yellow areas correspond to Indonesia.



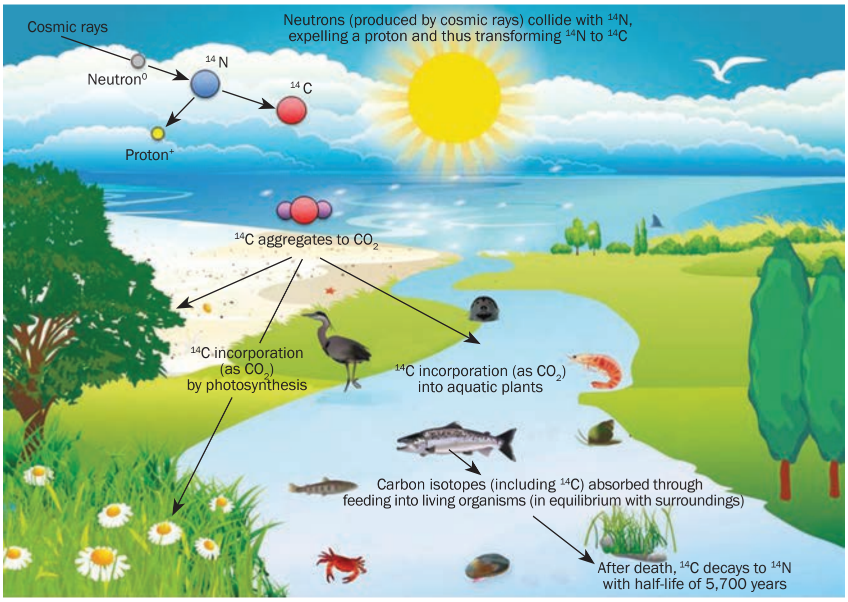


Figure 3: As shown in this schematic diagram of the radiocarbon cycle, radiogenic ^{14}C is produced in the atmosphere by the collision of high-speed neutrons (produced by cosmic radiation) with nitrogen (^{14}N). The traces of ^{14}C are in-corporated into carbon dioxide, which is assimilated into plants by photosynthesis and into animals (e.g. shells of molluscs) via respiratory and metabolic pathways. After death, the lifelong exchange of carbon with the environment suddenly stops, resulting in slow radioactive decay of ^{14}C, making it possible to determine the age of materials by radiocarbon dating. Illustration by M. S. Krzemnicki, using an artwork template from Inland Fisheries Ireland (www.somethingfishy.ie/resources/image_resources/image_estuary_food_web.jpg).

So far, the world's oldest dated pearl was recovered in the Middle East, and the stratigraphic layer in which it was found was attributed by Charpentier et al. (2012) to be around 7,500 years old. Szabo et al. (2015) dated the material surrounding a pearl found in Australia to more than 2,000 years old. In both cases, the pearl itself was not dated, and in recent years there have been only a few studies on radiocarbon age dating of historic pearls. For example, Krzemnicki and Hajdas (2013) performed age dating on historic and modern pearls. Recently, Zhou et al. (2017) obtained radiocarbon ages in the 16th century for pearls that reportedly came from the Venezuelan island of Cubagua in the Caribbean Sea, which supported the pre- to early Columbian era assumed for these pearls. Advances in testing and future archaeological finds will contribute to this area of pearl research by providing further evidence for the fishing and trade of pearls since ancient times in diverse regions of the globe (Kunz and Stevenson, 1908; Donkin, 1998).

Radiocarbon Age Dating

The underlying principle of the radiocarbon method is the constant production of radiogenic ^{14}C in the atmosphere by the interaction of secondary cosmic rays with nitrogen. The collision of high-speed neutrons produced by cosmic radiation with the nucleus of nitrogen results in the capture of a neutron and the expulsion of a proton, thus transforming the ^{14}N isotope into the radionuclide ^{14}C. The radiocarbon, present only in trace amounts in the atmosphere (about 1 atom per 1,012 atoms of carbon) combines with atmospheric oxygen and forms radioactive carbon dioxide (Figure 3), which is then incorporated into plants by photosynthesis and subsequently into animals via respiratory and metabolic pathways (Bowman, 1990; McConnaughey et al., 1997; Hajdas, 2008). As a consequence, the radiogenic ^{14}C is incorporated into the endo- or exoskeletons (e.g. bones or shell structures) of animals (Hajdas, 2008; Douka et al., 2010).

After death, the lifelong exchange of carbon with the environment suddenly stops, resulting in a slow radioactive decay of ^{14}C in the dead plants and animals. By measuring the ratio of radiogenic and stable carbon isotopes ($^{14}C/^{12}C$), it is thus possible to determine their age. The so-called half-life of ^{14}C (that is, the time at which only half of the original ^{14}C still is present in a sample and, as such, represents the constant rate of decay over time) is about 5,700 ± 40 years (Godwin, 1962). As a consequence, the maximum age dating reliably possible with this method is up to approximately 50,000 years before present (BP).

The radiocarbon age dating method, first described by Nobel laureate Willard Libby in the 1940s (Libby, 1946; Arnold and Libby, 1949) has been applied since then as a standard tool in numerous scientific disciplines and has gained much interest of the public, especially in relation with archaeological studies of Egyptian pharaohs and the prehistoric mummy 'Ötzi' from Austria (Bonani et al., 1994). Although it is applicable to only a very short period in geological terms, the

Figure 4: These 14 pearl samples (71742_A–N; approximately 2–8 mm diameter) from the Cirebon shipwreck were examined for this study. They are partially abraded around their drill holes and show some brown to grey colour alterations. Photo by Luc Phan, SSEF.

method has proven quite useful for dating organic matter (trees, tissues, etc.) and carbonaceous materials such as charcoal and biomineralization products, including corals (Adkins et al., 2002), shells (Berger et al., 1966; Hänni, 2008; Douka et al., 2010; Hainschwang et al., 2010) and pearls (Krzemnicki et al., 2009; Krzemnicki and Hajdas, 2013; Zhou et al., 2017).

A pearl is a calcium carbonate ($CaCO_3$) concretion formed by biomineralization processes in a mollusc—very much the same processes as for shell (exoskeleton) formation. As such, pearls (and shells) contain carbon, mainly the stable isotope ^{12}C (as well as ^{13}C) but also a small fraction of radiogenic ^{14}C. The carbon used for the biomineralization of pearls and shells mainly originates from two very different carbon pools: (1) oceanic dissolved inorganic carbon; and (2) respiratory CO_2, mainly stemming from food metabolism (Tanaka et al., 1986; Gillikin et al., 2007; Douka et al., 2010). As such, the so-called marine reservoir age effect may distinctly affect the resulting ^{14}C ages of shells and pearls, especially in areas with upwelling of 'old' water. Hence, a correction is required to take into account the geographic location of the sample. For a more detailed discussion of this issue, see Rick et al. (2005), McConnaughey and Gillikin (2008), Douka et al. (2010), Krzemnicki and Hajdas (2013) and references therein.

Samples and Methods

For this study, we investigated 14 pearls (nos. 71742_A–71742_N) from the Cirebon shipwreck (Figures 1 and 4) that weighed 0.14–0.85 ct and measured approximately 2–8 mm in diameter. They were round to button-shaped and baroque, and all showed a drill hole, indicating that they were originally at least partially strung on strands. This is further supported by the presence of abrasion marks around the drill holes, characteristic for pearls strung tightly in a row. The colour of the pearls ranged from white to light cream, some with brownish and greyish alterations (Figure 4) presumably due to oxidation of adjacent metallic material. Even after a prolonged period on the ocean floor, most of the pearls showed at least partially a soft nacreous lustre with some white dull weathered spots and patches.

All 14 pearls were analysed routinely by X-radiography (Faxitron unit) and X-ray luminescence (cf. Hänni et al., 2005), as well as by energy-dispersive X-ray fluorescence spectroscopy using a Thermo Quant'X instrument. We then selected four pearls (71742_A, B, I and J) for X-ray computed microtomography (micro-CT) analysis using a Scanco µCT-40 scanner. For radiocarbon age dating, we chose the three smallest pearls (71742_L, M and N). From each sample, ~8 mg of calcium carbonate was extracted either by abrading or chipping off nacre fragments from the pearls, which was facilitated by their slightly altered surface condition. However, based on this and more recent experiments, we now can perform quasi-non-destructive radiocarbon age dating with as little as ~2 mg (0.01 ct) of nacre taken from the drill hole, thus not affecting the outer surface of the pearl (Krzemnicki, 2017).

The calcium carbonate samples were washed in ultrapure water and leached to remove the surface layers (Hajdas et al., 2004). After the leaching of about 20% (by weight) of the original sample, approximately 6.4 mg of pearl material was placed in a gas bench tube and flushed with a flow of helium gas, then dissolved in concentrated phos-

Table I: Results of ^{14}C analyses of three saltwater pearls from the Cirebon shipwreck in the Java Sea.[a]

Sample	^{14}C age (BP, ±1σ)	$\delta^{13}C$ (‰)[b]	$F^{14}C$ (±1σ)[c]	Calendar age (calAD)	
				68.2% prob.	95.4% prob.
71742_L	1,529 ± 25	–0.7 ± 1.0	0.8267 ± 0.0026	878–1042	780–1130
71742_M	1,493 ± 25	–1.9 ± 1.0	0.8304 ± 0.0026	902–1072	816–1170
71742_N	1,508 ± 25	–3.2 ± 1.0	0.8288 ± 0.0026	894–1058	804–1156

[a] Abbreviations: BP = before present; prob. = probability.

[b] The isotopic signature $\delta^{13}C$ is a measure of the ratio of stable isotopes $^{13}C/^{12}C$, and is reported in parts per thousand (per mil, ‰).

[c] The fraction of modern radiocarbon ($F^{14}C$) is the conventional way of displaying the so-called 'bomb peak' in a radiocarbon vs. known age diagram for post-1955 events.

phoric acid (85%) and transferred to a graphitization system (Wacker et al., 2013). The graphite was then pressed into targets (cathodes), and the $^{14}C/^{12}C$ ratio was measured using the Mini Carbon Dating System (MICADAS; see Synal et al., 2007) at the Swiss Federal Institute of Technology, ETH Zürich, Switzerland. This optimized accelerator mass spectrometer (AMS) is characterized by a high yield and superior stability, thus enabling radiocarbon measurements at highest precision. Different from other mass spectrometer designs, the ions formed in the AMS ion source are negative, thereby filtering out ^{14}N, which is an isobar of ^{14}C. Then the ions are accelerated, reaching very high kinetic energies and resulting in a high resolving power for separating a rare isotope from an abundant neighbouring mass, such as ^{14}C from ^{12}C. Moreover, a suppression of molecular isobars (e.g. ^{13}CH and $^{12}CH_2$) is achieved by passing the beam through a stripper gas. Finally, the ^{14}C atoms are detected by a gas ionization system (Synal et al., 2007).

After correction for blank values and fractionation ($\delta^{13}C$), the measured $^{14}C/^{12}C$ concentration was used to calculate conventional ^{14}C ages (Stuiver and Polach, 2016). For all samples, the calculated ^{14}C age BP was corrected by applying a marine reservoir correction (delta R = 89 ± 70) that was based on values for the Java Sea location (Reimer and Reimer, 2001, and references therein). These were estimated (weighted mean) based on 10 data points in the vicinity of the sampling site. The corrected ^{14}C ages were then calibrated using the Marine13 curve of Reimer et al. (2013).

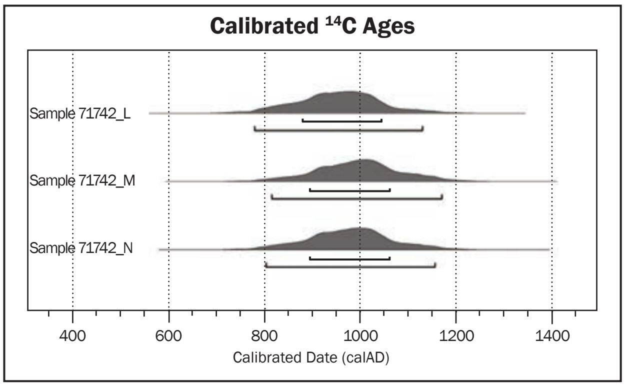
Results and Discussion

Based on their X-radiographs, trace-element composition (cf. Gutmannsbauer and Hänni, 1994) and lack of luminescence to X-rays (cf. Hänni et al., 2005), the samples studied for this report were all saltwater natural pearls. The radiography and micro-CT scans (on pearls 71742_A, B, I and J) further revealed that their internal structure mainly consisted of fine ring structures typical of natural pearls.

Table I summarizes the results of radiocarbon age dating of the three pearls (71742_L, M and N). They all show very consistent ^{14}C ages and similar calibrated ages of 780–1170 AD (95.4% probability) or 878–1072 AD (68.2% probability) using the Marine13 curve (Reimer et al., 2013; Figures 5 and 6). The more precise mean value of 1,510 ± 15 BP results in a calendar age of 806–1151 AD (Figure 7). This relatively wide range in calendar age is due to uncertainty for the reservoir age correction.

The calculated age of the pearls, corresponding approximately to the end of the 10th century, correlates well with the age stipulated for the coins, pottery and other artefacts found in the shipwreck (Liebner, 2014). It places the sinking of the historic merchant vessel at the time of upheaval in China

Figure 5: The three investigated pearls (71742_L, M and N) from the Cirebon shipwreck all show very similar calibrated ages corresponding to the late 10th century AD. The bracketed ranges represent 68.2% and 95.4% probabilities for the pearls' ages.



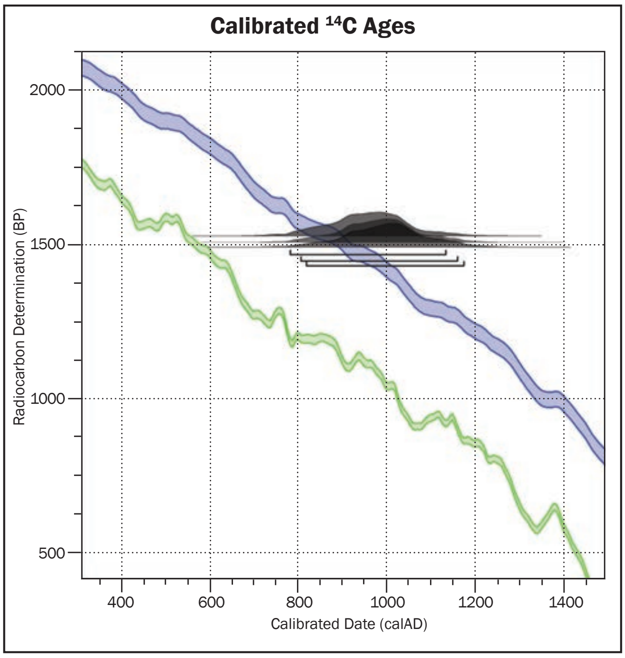


Figure 6: The calibrated ages of the three investigated pearls from the Cirebon shipwreck were derived from the radiocarbon ages using OxCal 4.2 software (Ramsey and Lee, 2013). The purple band is the marine calibration curve (Marine13, Reimer et al., 2013), and the green band is the atmospheric curve (IntCal13, Reimer et al., 2013).

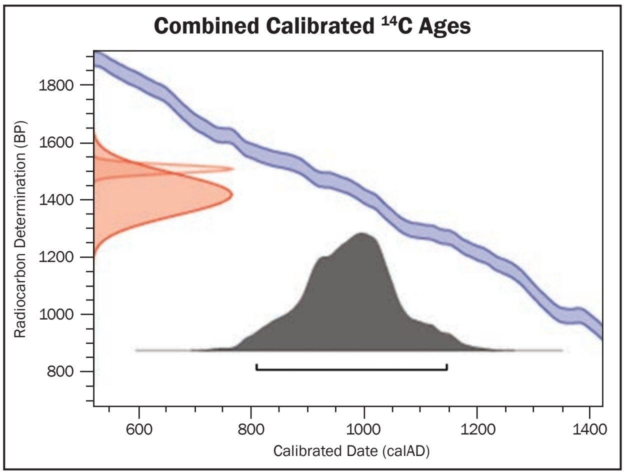


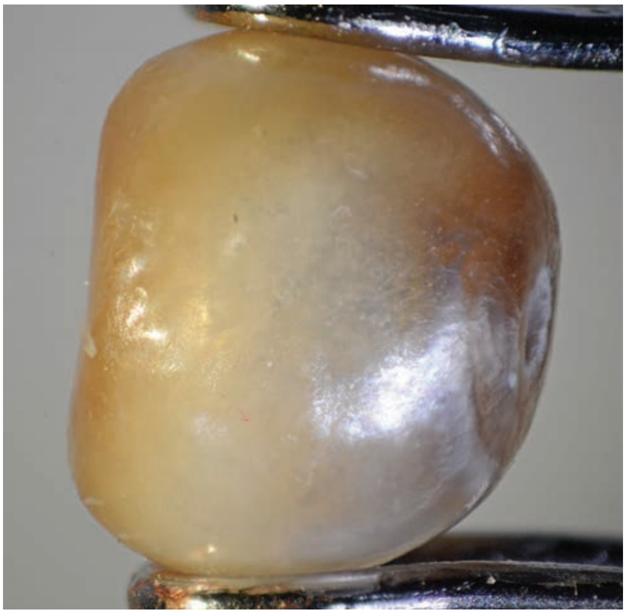
Figure 7: The combined calibrated ages obtained from the three Cirebon pearls yield a range of 806–1151 AD at 95.4% probability. The red peaks represent the uncalibrated radiocarbon age (as measured and corrected for regional reservoir age) and the dark grey peak shows the distribution of calendar ages.

called the 'Five Dynasties and Ten Kingdoms' (ca. 907–960 AD), which was also a time of extensive maritime trade in Southeast Asia.

These pearls likely originated from the Persian Gulf or the Gulf of Mannar (between India and Sri Lanka), both known since ancient times as sources of saltwater natural pearls (from *Pinctada radiata*; see Hornell, 1905; Carter, 2005). This assumption is mostly related to their size, bearing in mind that other molluscs also produced (larger) nacreous pearls during the same period (e.g. *P. margaritifera* in the Red Sea and *P. maxima* in Southeast Asia; Southgate and Lucas, 2008). The thousands of glass fragments and several unbroken blue and green glass objects found in the Cirebon shipwreck undoubtedly originated from the Islamic Middle East (present day Iran or Iraq and Syria; H. Bari, pers. comm., 2017). This indicates extensive trade in Southeast Asia along maritime routes (or a 'maritime silk route') at that time (Liebner, 2014; Manguin, 2017), of which the Cirebon merchant vessel was a part. This also supports a Persian Gulf origin for the pearls (H. Bari, pers. comm., 2017).

The partly abraded and brown-to-grey alterations around the drill holes of these pearls (Figure 8) suggest that they might have been in use for some time, strung on strands or set with metal linings in jewellery before they sank in the vessel with the rest of its cargo, including Sri Lankan sapphires (Henricus, 2014) and other gems (e.g. red garnet and quartz) of probable Sri Lankan, East African or Malagasy origin (H. Bari, pers. comm., 2017).

Figure 8: Pearl 71742_A (0.85 ct) shows brownish colour alteration around its drill hole (visible on the right side), as would be expected from a setting with a metal lining that has oxidized. The abrasion features adjacent to the drill hole are consistent with wear marks commonly encountered on pearls that are tightly strung on wire or fibre strands. Photo by M. S. Krzemnicki, SSEF.



Conclusions

This study is the first to document radiocarbon age dating, along with gemmological testing, carried out directly on historic pearls dating back to the 10th century. Previous research on historic pearls, including the 2,000-year-old Brremangurey pearl from Western Australia (Szabo et al., 2015) or the 7,500-year-old Umm al-Quwain pearl from UAE (Charpentier et al., 2012), derived their ages by using associated materials found at the archaeological sites, rather than directly dating the pearls themselves.

By using the highly sensitive MICADAS system at the Ion Beam Physics Laboratory at ETH Zürich, it was possible to analyse very minute portions of the Cirebon pearls. The radiometric age dating of the three samples gave homogeneous results corresponding approximately to the end of the 10th century, closely matching the age stipulated for the shipwreck based on Chinese pottery and coins.

This study, and further age-dating experiments on pearls, also have resulted in a refined sampling process that allows us to work with tiny amounts of nacre powder (~2 mg) taken from the drill hole without any damage to the outer surface of a pearl. Thus, radiocarbon age dating can be considered a quasi-non-destructive test when following our sampling protocol. This has opened up new possibilities for research on historical biogenic objects and artefacts of significance to archaeology and cultural heritage.

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Acknowledgements

The authors thank their colleagues at SSEF, and also Hubert Bari (pearl expert and author of the books *Pearls* and *The Pink Pearl*), for valuable comments and assistance. The peer reviewers also added helpful information and suggestions to the article.

Thank You, Guest Reviewers

The following individuals served as guest reviewers during the past publication year. A special thanks is extended to each one of them for lending their expertise to reviewing manuscripts submitted to *The Journal*. Together with the Associate Editors, these individuals have enhanced the quality of *The Journal* through their knowledge and professionalism.

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