VARIATION IN RADIOCARBON AGE DETERMINATIONS FROM THE CRYSTAL RIVER ARCHAEOLOGICAL SITE, FLORIDA

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ABSTRACT. Archaeologists interested in radiocarbon dating shell middens sites express concern regarding the accuracy of shell dates and how such determinations should be interpreted. This article discusses the problem of dating shells from sites in the southeastern United States. New results are presented comparing shell, bone, and soil-charcoal age determinations from the Crystal River site, located along the west-central Gulf Coast of Florida. Crystal River is a large multimound site whose occupants engaged in long-distance exchange throughout eastern North America during the Woodland period (~1000 BC to AD 1050). In the summer of 2012, test units were excavated in several contexts at the site, including both mounds and occupation areas. Samples were collected for 14C dating, which were then processed at the University of Georgia Center for Applied Isotope Studies. This article focuses on samples from the stratified shell midden, from which it was hoped to construct a local correction for marine shell that could be used to date other contexts. The soil-charcoal and bone collagen from these samples have very similar ages (bone samples ranging from about 100 cal BC to cal AD 530 and soil-charcoal from cal AD 345 to 560); however, the shell samples collected from the same stratigraphic units are significantly older than the terrestrial dates (ranging from 1300 to 390 cal BC). The difference in calibrated ages between organic materials and the shells ranges between 560 to 1140 yr. This phenomenon cannot be explained solely by the marine reservoir effect. It appears that all the shell samples formed in mixed marine (~50−60%) contexts, as indicated by the stable isotope ratios and the amount of atmospheric carbon remaining in the samples. The age of the shell samples cannot be used to date archaeological events as they are influenced not only by the marine reservoir effect, but also the local hardwater effect, which makes them significantly older.

INTRODUCTION

Archaeologists in coastal and other aquatic regions around the world rely heavily on radiocarbon dating of shell for building artifact, site, and regional chronologies. Shell provides a number of advantages over other sources of carbon: shell is typically abundant on coastal sites; most marine mollusks have relatively short lifespans; shell is not contaminated by modern vegetation decay or rootlets; and, finally, larger shells arguably do not move as readily through the stratigraphic column as small artifacts and ecofacts (Kennett et al. 2002; Thomas 2008:346). However, shell is notoriously difficult to date accurately because of reservoir effects. For this reason, many archaeologists have traditionally avoided dating shell, despite its advantages.

The use of marine and freshwater shell for 14C dating has become increasingly popular as techniques for correcting and calibrating 14C dates on shell have become more sophisticated and as significant strides have made in understanding spatial and temporal variation in the local reservoirs and other sources of bias (Stuiver and Braziunas 1993; Stuiver et al. 1986, 1998). Such studies have resulted in an increased confidence in the use of marine and freshwater shell to date archaeological materials in some coastal areas, including parts of North America (e.g. Erlandson et al. 1996; Kennett et al. 1997; Deo et al. 2004; Thomas 2008; Thomas et al. 2013). However, in many other areas the dating of shell remains problematic because local reservoir effects are poorly understood. This study describes efforts to date shell from one such region, the eastern Gulf of Mexico, focusing specifically on the archaeological site of Crystal River.

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SITE DESCRIPTION

The Crystal River site (8CI1) is located on Florida’s west-central coast (Figure 1), adjacent to the river of the same name. Crystal River flows approximately 9 km from its source at a series of springs to its terminus at the Gulf of Mexico. The archaeological site lies about 4 km from the river’s source.

The Crystal River site extends over 8 ha and includes one large, flat-topped, ramped mound (Mound A); two smaller platform mounds, one also ramped (Mound H) and the other possibly so (Mound K); two burial mounds, one discrete (Mound G) and the other actually a complex of earthworks (mounds C–F); three limestone boulders (stelae 1–3), and a large comma-shaped midden (Weisman 1995; Pluckhahn et al. 2010) (Figure 2). The work of C B Moore (1903, 1907, 1918; see also Greenman 1938) in the early 20th century established the site’s fame as one of the largest mound complexes on the Gulf Coast and the southernmost expression of what would later become known as the Hopewell Interaction sphere, a network of exchange of exotic goods that linked distant communities across eastern North America. Later investigations by R Bullen (1951, 1953, 1966) and G Willey (1948a,b, 1949), among others (e.g. Smith 1951), developed the site’s general chronological placement in the Woodland period, from around 1000 BC to AD 1050, based mainly on relative artifact chronologies.

Contemporary archaeological work at Crystal River has been limited and modest in scope. Prior to recent work, only three new radiocarbon dates (Katzmarzyk 1998) had been obtained from the site in more than 40 yr; thus, these few were the only dates from the site conducted according to contemporary protocols and with acceptable standard deviations. The Crystal River Early Village
Archaeological Project (CREVAP) is a 3-yr NSF-funded study of the dynamics of competition and cooperation in early villages, using Crystal River as a case study (Pluckhahn et al. 2010). Given the paucity of modern archaeological investigations and reliable absolute dates, mounds, a fundamental priority for CREVAP is more precise and accurate dating of Crystal River’s mounds and middens. Moore (1903:379) described the midden at Crystal River as a “low, irregular shell deposit” that curved east from Mound A, “extending for some distance along the riverbank.” Willey (1949:41) noted with greater specificity that the midden extended over 1000 ft (304.8 m) in length and 100 ft (30.5 m) in width. Willey described the composition of the midden as “shells and rich black midden.” Neither Moore nor Willey conducted excavations in the midden. However, Bullen excavated a few test units in the area in the 1950–1960s. His work has never been adequately reported, but his notes (on file at the Florida Museum of Natural History, Gainesville) indicate that midden extends to a depth of more than 84 inches (213 cm). Much of the midden was at least partially destroyed for
the in-filling of an adjacent lagoon in the 1960s. However, substantial areas of the midden are well preserved.

This study reports $^{14}$C samples from Trench 1 (Figure 2), excavated into the shell midden deposits in large part to establish a baseline chronology for the occupation of Crystal River. We intended to date shell and other materials from the same levels in the stratified midden deposits in order to develop a correction factor for shell that could be used for dating other contexts at Crystal River, especially those such as mounds where shell was often the only recovered datable material.

Trench 1 was located east of Mound K on one of the highest and best preserved portions of the midden (Figure 3). The trench, measuring $1 \times 4$ m long and oriented east to west, was excavated with a combination of natural and arbitrary 10-cm levels in $1 \times 1$ m sections labeled test units 1–4. Test units 3 and 4, at the eastern end of the trench, encountered one of Bullen’s older excavation units and were not excavated below a depth of 40 cm. Test units 1 and 2 were excavated to 142 cm below the ground surface (152 cm below datum [cmbd]), where water was encountered at high tide, thus preventing deeper excavation.
Figure 4 is a profile drawing of the stratigraphy in Trench 1. Several clear stratigraphic breaks are indicated by changes in soil color and texture and by the relative abundance of inclusions, principally oyster (*Crassostrea virginica*) shell. Stratum IV was particularly well differentiated by dark soil with a significantly reduced quantity of oyster shell.

![Figure 4 Stratigraphic profile of Trench 1 at the Crystal River site](image)

Several features, probably representing post molds or small pits, originate at this stratum. Noticeably absent is any evidence for appreciable mixing of stratigraphic layers apart from these features. The stratigraphic integrity of the trench is further indicated by the distribution of certain classes and types of artifacts. In Unit 1, for example, conch shell tools were restricted to levels 1–5 (10–52 cmbd), flaked stone artifacts were limited to levels 1–9 (10–92 cmbd), and Deptford Check Stamped pottery (the earliest clear diagnostic type at Crystal River) was found only in levels 13–15 (132–152 cmbd).

We excavated a 25 × 25 cm column sample (Column 1) midway along the western wall of Trench 1 (Figure 3), in an area where no features (which could potentially mix stratigraphy) were apparent. The column sample was initially excavated in 2-cm levels, but when this proved impractical because of the density of shell we switched to 4-cm levels. The soil from each level was collected without screening. To maximize stratigraphic integrity and minimize cross-sample contamination, excavation tools were washed with distilled water before taking each sample. The same precautions were observed in the laboratory when subsamples were removed for 14C dating.

**RADIOCARBON DATING**

We 14C dated three types of materials from Trench 1: terrestrial mammal bone, oyster shell, and charcoal from soil samples. Small fragments of charcoal are abundant in the midden at Crystal River, allowing us to date very small samples of soil. As noted above, samples were dated of different materials from the same, or equivalent, stratigraphic levels.

The mammal bone samples were brushed to remove surrounding soil, washed, and ultrasonically cleaned in deionized water, then dried in an oven at 60°C. The crushed bone was treated with
1N HCl at 4°C for 24 hr. The residue was filtered, rinsed with deionized water, and treated with 0.1N NaON to remove contamination from humic acids. The collagen was then rinsed with deionized water and diluted HCl and deionized water again and under slightly acid condition (pH = 5), heated at 80°C for 16 hr to dissolve collagen, and with humic substances remaining in the precipitate. The collagen solution is then filtered to isolate pure collagen and dried out. The dried collagen was combusted at 575°C in an evacuated/sealed Pyrex® ampoule in the presence of CuO. The CO₂ and nitrogen have been cryogenically separated for analyses.

The oyster shell samples were etched in diluted hydrochloric acid at room temperature in an ultrasonic bath to remove the surface contaminants and outer layer most susceptible to diagenesis, then samples were rinsed in deionized water. The prepared shell samples were dried in an oven at 105°C and crushed to powder, then converted to CO₂ by reaction with 100% phosphoric acid under vacuum. Subsamples were used to measure δ¹³C/¹²C ratios and the remaining samples were converted to graphite.

The soil samples were saturated with charcoal fragments, which were manually picked free of roots and shell fragments, and then treated with 1N HCl acid at 90°C for 1 hr, rinsed with deionized water, and treated with 0.1N sodium hydroxide for 15 min at the same temperature to remove humic acids. They were then rinsed again with deionized water. Finally, the samples were treated with 1N HCl, rinsed, and dried at 105°C. The cleaned samples were combusted at 900°C in an evacuated/sealed quartz ampoule in the presence of CuO. The CO₂ was cryogenically separated for analyses.

For accelerator mass spectrometry (AMS) analysis, the cleaned CO₂ was catalytically converted to graphite using the method described in Cherkinsky et al. (2010). Graphite ¹³C/¹²C ratios were measured using the 0.5MV Pelletron AMS instrument at the University of Georgia. The sample ratios were compared to the ratio measured from oxalic acid standard OXI (NBS-4990) to calculate ¹⁴C. The obtained ¹⁴C ages were converted to calendar dates by using the calibration program CALIB 6.0 with the IntCal09 curve (Reimer et al. 2009) for soil and bone collagen samples and the mixed marine and atmosphere curve for shell samples.

RESULTS AND DISCUSSION

In total, 18 samples were dated from Trench 1 (Table 1): 10 soil-charcoal samples, 4 terrestrial mammal bones (collagen fraction), and 4 oyster shell samples. A comparison of ¹⁴C dates for the soil-charcoal and bone collagen samples shows good agreement between them in most cases, with only one significant outlier of collagen dating to 171–37 BC. The bone collagen samples ranged from about 100 cal BC to cal AD 530 and soil-charcoal from cal AD 345 to 560. However, the shell samples collected from the same stratigraphic units are significantly older than the terrestrial dates, ranging from 1300 to 390 cal BC. The difference in calibrated ages between organic materials and the shells vary between 560 and 1140 yr. Looking at paired bone and shell samples (n = 4), the differences in the measured ¹⁴C age BP ranged from 530 to 1850 yr, with a mean of 1258 yr. The differences between paired soil-carbon and shell samples (n = 3) ranged from 920 to 1380, with a mean of 1163 yr. This phenomenon cannot be explained by the marine reservoir effect, which does not exceed 150 yr for the interior portion of the Georgia Bight (Thomas 2008).

As noted above, archaeologists working elsewhere in southeastern North America have reported greater success in dating shell. Most pertinent for our study, Thomas (2008) ¹⁴C dated 11 pairs of marine shell and charcoal samples from sites on St. Catherines Island, on the Atlantic coast of Georgia. The shell samples ranged from 90 to 500 yr older than the paired charcoal samples. The mean age differential between the charcoal and shell dates was 320 yr, far less than the discrepancies between shell and terrestrial samples at Crystal River.
Table 1  Radiocarbon and calendar age (2σ probability) of different materials from Crystal River Site.

<table>
<thead>
<tr>
<th>UGAMS #</th>
<th>Sample ID</th>
<th>Provenience</th>
<th>Depth (cm)</th>
<th>Material</th>
<th>δ¹³C,‰</th>
<th>¹⁴C age, yr BP ± pMC ± Calibrated age 2σ</th>
</tr>
</thead>
<tbody>
<tr>
<td>12943</td>
<td>8CI1SS770C</td>
<td>Column 1, Level 15</td>
<td>40–42</td>
<td>Charcoal</td>
<td>−24.6</td>
<td>1490 ± 83.0 0.24 cal AD 540–635</td>
</tr>
<tr>
<td>12135s</td>
<td>8CI1FS639s</td>
<td>Unit 1, Level 5</td>
<td>42–52</td>
<td>Charcoal</td>
<td>−22.7</td>
<td>1540 ± 82.6 0.23 cal AD 432–576</td>
</tr>
<tr>
<td>12136</td>
<td>8CI1FS639B</td>
<td>Unit 1, Level 5</td>
<td>42–52</td>
<td>Collagen</td>
<td>−21.4</td>
<td>1610 ± 81.8 0.23 cal AD 405–535</td>
</tr>
<tr>
<td>12135</td>
<td>8CI1FS639S</td>
<td>Unit 1, Level 5</td>
<td>42–52</td>
<td>Shell</td>
<td>−4.2</td>
<td>2730 ± 71.2 0.21 cal BC 765–417</td>
</tr>
<tr>
<td>12944</td>
<td>8CI1SS788C</td>
<td>Column 1, Level 20</td>
<td>50–52</td>
<td>Charcoal</td>
<td>−23.7</td>
<td>1730 ± 80.7 0.23 cal AD 250–382</td>
</tr>
<tr>
<td>12945</td>
<td>8CI1SS795C</td>
<td>Column 1, Level 25</td>
<td>60–62</td>
<td>Charcoal</td>
<td>−24.2</td>
<td>1720 ± 80.8 0.23 cal AD 250–392</td>
</tr>
<tr>
<td>12946</td>
<td>8CI1SS799C</td>
<td>Column 1, Level 29</td>
<td>71–75</td>
<td>Charcoal</td>
<td>−23.5</td>
<td>1650 ± 81.4 0.24 cal AD 264–510</td>
</tr>
<tr>
<td>12947</td>
<td>8CI1SS804C</td>
<td>Column 1, Level 33</td>
<td>87–91</td>
<td>Charcoal</td>
<td>−21.5</td>
<td>1720 ± 80.7 0.23 cal AD 250–392</td>
</tr>
<tr>
<td>12133</td>
<td>8CI1FS660B</td>
<td>Unit 1 Level 9</td>
<td>82–92</td>
<td>Collagen</td>
<td>−11.5</td>
<td>2070 ± 77.3 0.23 171–37 cal BC</td>
</tr>
<tr>
<td>12134s</td>
<td>8CI1FS660s</td>
<td>Unit 1 Level 9</td>
<td>82–92</td>
<td>Shell</td>
<td>−4.9</td>
<td>2600 ± 72.3 0.21 666–358 cal BC</td>
</tr>
<tr>
<td>12948</td>
<td>8CI1SS807C</td>
<td>Column 1, Level 36</td>
<td>99–103</td>
<td>Charcoal</td>
<td>−20.4</td>
<td>1750 ± 80.4 0.23 cal AD 231–382</td>
</tr>
<tr>
<td>12949</td>
<td>8CI1SS812C</td>
<td>Column 1, Level 39</td>
<td>111–115</td>
<td>Charcoal</td>
<td>−20.4</td>
<td>1760 ± 80.3 0.23 cal AD 214–381</td>
</tr>
<tr>
<td>12950</td>
<td>8CI1SS814C</td>
<td>Column 1, Level 41</td>
<td>119–123</td>
<td>Charcoal</td>
<td>−20.4</td>
<td>1830 ± 79.6 0.24 cal AD 92–245</td>
</tr>
<tr>
<td>12518</td>
<td>8CI1FS695B</td>
<td>Unit 1, Level 13</td>
<td>122–132</td>
<td>Collagen</td>
<td>−21.7</td>
<td>1690 ± 81 0.23 cal AD 259–413</td>
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<tr>
<td>12517</td>
<td>8CI1FS695S</td>
<td>Unit 1, Level 13</td>
<td>122–132</td>
<td>Shell</td>
<td>−4.9</td>
<td>3210 ± 67.1 0.2 1406–1265 cal BC</td>
</tr>
<tr>
<td>12519</td>
<td>8CI1FS696S</td>
<td>Unit 1, Level 14</td>
<td>132–142</td>
<td>Shell</td>
<td>−5.8</td>
<td>2710 ± 71.4 0.21 768–404 cal BC</td>
</tr>
<tr>
<td>12520</td>
<td>8CI1FS696B</td>
<td>Unit 1, Level 14</td>
<td>132–142</td>
<td>Collagen</td>
<td>−21.8</td>
<td>1860 ± 79.3 0.22 cal AD 85–222</td>
</tr>
</tbody>
</table>
Marine upwelling does not appear to account for the differences in the results between St. Catherines Island and Crystal River. Although located in a tidal estuary, Crystal River is located ~5 km upstream from the Gulf of Mexico, and is well protected from Gulf currents by a series of islands and estuaries (see Figure 1). Thus, like St. Catherines Island, we do not think that upwelling has a significant effect (Thomas et al. 2013). Unlike St. Catherines Island, the waters of Crystal River run through a limestone substrate (Cooke 1945; Pliny et al. 1988). It is likely that its waters carry dissolved carbonates from limestone “old carbon,” which has little or no 14C. This phenomenon, known as the hardwater effect, has been documented in other areas of the world with carbonaceous geology (Bezerra et al. 2000; Dye 1994; Gischler et al. 2008) but not, to our knowledge, for North America. Describing the hardwater effect as it relates to the dating of molluscan shells, Douka et al. (2010:21–2) observe that these “species absorb dissolved carbon dioxide (CO$_2$) or bicarbonates (HCO$_3$)” leached out from limestone areas (dead-carbon sources), which make them exhibit reduced activity and hence display an older radiocarbon age.” Following Forman and Polyak (1997), they further suggest that “the effect is larger in molluscan shells growing in localities (a) with restricted water circulation (b) where there is considerable mixing of fresh and oceanic water, (c) where the geological substrate is highly carbonaceous and (d) in areas with high abundance of terrestrial organic matter.” All of these compounding conditions are applicable to Crystal River. The fact that the low gradient of the continental shelf on the coast of the Gulf of Mexico leads to considerable mixing of fresh and oceanic water is confirmed by our analysis; stable isotope ratios and the amount of atmospheric carbon remaining in the samples indicate that they formed in mixed marine (~50–60%) contexts. The substrate at Crystal River is comprised of limestone; an auger test extended below the floor of Trench 1 encountered limestone within ~20 cm (at a depth of ~152 cm) and excavations elsewhere at the site encountered limestone within 1 m of the surface. Because the climate is humid subtropical, there is an abundance of terrestrial organic matter. Water circulation is not restricted today, but may have been in the past when sea levels were lower at Crystal River. It is difficult to estimate the dependency between terrestrial and shell sample ages, as there are currently only four pairs (Figure 5) of these kinds of samples. A preliminary regression equation, which allows to correct the age for this hardwater effect is as follows:

\[ y = -0.306x + 2556 \text{ yr}; \quad R^2 = 0.2378 \]

where \( y \) is the corrected for hardwater effect shell age and \( x \) is the 14C age of the shell. However, the variation between shell and mammal bone or soil charcoal is not clearly consistent in our limited sample, so the usefulness of this regression equation for the correction of shell dates must await additional research. This greater variability in shell dates from Crystal River relative to those from St. Catherines Island is difficult to interpret. Species habitat may play a role, as Douka et al. (2010) suggest, but this is poorly understood. All of the marine samples from Crystal River were oyster, but this species can tolerate a range of salinity. It is possible that the prehistoric residents of Crystal River gathered some oysters from areas more susceptible to the hardwater effect due to greater mixing of fresh- and seawater and others from areas where the effect was reduced, and that this variability is reflected in our samples.
CONCLUSIONS

This study observed clear differences in $^{14}$C ages for terrestrial and aquatic materials collected from the same levels for this site. The charcoal and bone collagen samples were consistently younger than the shell samples. The differences were not predictable, with shell samples ranging from 530 to 1850 yr older than paired bone samples and from 920 to 1380 yr older than paired soil-charcoal samples. The overall mean age differential between the charcoal/bone and shell dates was 1217 yr. There is no clear dependency between $^{14}$C ages of aquatic and terrestrial samples, so this study will be continued for the precise estimation of the hardwater effect at Crystal River.

The conditions accounting for the differential between terrestrial and marine samples are not unique to Crystal River. Other portions of the coast of the Gulf of Mexico, including most of peninsular Florida and the Yucatan Peninsula, are underlain by carbonaceous substrate. Archaeologists and geologists working in these and similar areas would do well to understand the local environment before depending on $^{14}$C dates on marine shell, despite the relative advantages the dating of this material may otherwise offer.

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REFERENCES

