DIFFERENCES IN ^{14}C AGE BETWEEN STRATIGRAPHICALLY ASSOCIATED CHARCOAL AND MARINE SHELL FROM THE ARCHAIC PERIOD SITE OF KILOMETER 4, SOUTHERN PERU: OLD WOOD OR OLD WATER?

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ABSTRACT. Consistently large differences occur in the calibrated ^{14}C ages of stratigraphically associated shell and charcoal samples from Kilometer 4, an Archaic Period archaeological site located on the extreme south coast of Peru. A series of nine shell and charcoal samples were collected from a Late Archaic Period (~6000–4000 BP) sector of the site. After calibration, the intercepts of the charcoal dates were ~100–750 years older than the paired shell samples. Due to the hyper-arid conditions in this region that promote long-term preservation of organic material, we argue that the older charcoal dates are best explained by people using old wood for fuel during the Middle Holocene. Given this "old wood" problem, marine shell may actually be preferable to wood charcoal for dating archaeological sites in coastal desert environments as in southern Peru and Northern Chile.

INTRODUCTION

Radiocarbon dating is the primary method for establishing pre-ceramic cultural chronologies in Peru. The two primary assumptions that underlie the ^{14}C technique are that 1) the initial ^{14}C content of same age samples is similar and 2) the ^{14}C content of samples is not altered in the post-depositional environment (Taylor 1987). Depending upon geographical and depositional context, all datable organic materials can have inherent problems (Dean 1978; Arundale 1981; Schiffer 1986; Hedges and Van Klinken 1992; Dye 1994). Determining and correcting for any disparities in ^{14}C content is essential for the comparability of ^{14}C dates and the accurate development of cultural chronologies in Peru and elsewhere.

In coastal Peru, charcoal and marine shell are the two primary material types available in archaeological sites for ^{14}C dating. Marine shell is often the most abundant and well preserved of these, but archaeologists working in the region are reluctant to date shell except in cases where it is the only material available (e.g. Sandweiss et al. 1989). This is because anomalies in marine shell ^{14}C values were documented in coastal Peru early in the development of the ^{14}C method (Rowe 1965). The legendary problems with shell dates in Peru are related to their old initial ages caused by upwelling and slow mixing of ^{14}C depleted deep ocean water with more recently formed surface waters. In some cases, the carbonate in deep ocean water is up to 1000 years older than carbonate in surface waters. Old carbonate is incorporated into the shell matrices of molluscs during growth, resulting in ages older than expected. This is known as the marine reservoir effect (Taylor 1987).

Archaeologists working in other regions commonly ^{14}C date marine shell and calibration procedures have become more sophisticated and accurate (Ingram and Southon 1996; Erlandson et al. 1996; Kennett et al. 1997). Calibration of marine shell ^{14}C dates is based on a spatial model of ^{14}C content of oceanic waters (Stuiver et al. 1986, 1998; Stuiver and Braziunas 1993). This model corrects for much of the variation in the marine ^{14}C reservoir, but regionally specific deviations also occur (ΔR). There is a significant range in ΔR values due to the complexities of upwelling and ocean circulation. In general, ΔR values are derived for a particular geographic region based on ^{14}C dates on a rela-

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tively small number of "pre-bomb" shells of known age. For instance, the ΔR value for Peru is 190 ± 40 years, an average calculated from ^{14}C measurements on only three known-age shells from a ~2000 km stretch of coastline (Taylor and Berger 1967; Stuiver and Braziunas 1993).

The primary assumption when using ΔR values is that they have remained the same through time. This assumption has been tested by comparing the ^{14}C dates of paired marine and terrestrial materials in natural and archaeological deposits (Southon et al. 1986, 1990; Alberto et al. 1986; Kennett et al. 1997). In some temporal and spatial contexts, the estimated ΔR values appear to work well (Southon; Rodman and True 1986; Kennett et al. 1997), however there appear to be certain places and intervals of time when these values are unsatisfactory. For instance, shell and charcoal pairs from Daisy Cave, located on California's Channel Islands, indicate that ΔR values fluctuated along the southern California Bight during the Holocene (Kennett et al. 1997). This is not surprising, given oscillations in oceanographic circulation and upwelling that are evident in the region during the late Quaternary (Kennett and Ingram 1995; Kennett and Kennett 2000).

In this study, we have ^{14}C dated a series of shell and charcoal samples (*N=18*) from the same stratigraphic contexts at the archaeological site of Kilometer 4, located on the southern coast of Peru near the modern-day town of Ilo (Wise 1999). This deeply stratified site has multiple components dating to the Early and Middle Holocene, including distinctive domestic features (house floors and terraces), cemeteries, and extensive, deeply stratified shell middens (Wise 1999). Prehistoric materials are well preserved at this location due to the dry conditions along this stretch of coastline. The study was designed to establish a ^{14}C chronology for a roadcut exposure known as the railroad profile (Figure 1) and to refine the ΔR value for the Middle Holocene along this stretch of coast. Although long-term marine climate records are not available for Peru, variations in upwelling certainly occur due, in part, to the periodic affect of El Niño/southern oscillation (ENSO). Therefore, it is possible that ΔR values have fluctuated during the Holocene in a similar fashion to those in southern California.

METHODS

Shell and charcoal samples were collected from the railroad profile at Kilometer 4, a well-preserved midden deposit exposed in a roadcut on the southwestern side of the site (Figure 1). In 1994, this ~2 m section was cleaned so that shell and charcoal samples could be extracted from intact strata for ^{14}C dating. A series of 9 paired shell and charcoal samples (*N=18*) were taken from the occupational sequence of cultural strata. In each stratigraphic layer the shell and charcoal samples were taken in close proximity (no more that 3 cm apart).

Charcoal samples were selected for radiocarbon dating in the archaeological laboratories at UC Santa Barbara and sent to the Center for Accelerator Mass Spectrometry (CAMS) at the Lawrence Livermore National Laboratory for ^{14}C dating (Davis et al. 1990). Prior to analysis, charcoal samples (1–2 mg) were rinsed sequentially in weak acid (1N hydrochloric acid) and base (1N sodium hydroxide), ending with a weak acid rinse to remove CO_2 absorbed during the alkaline bath. The procedure removes any adhering organic acids and secondary carbonate. Organic samples were then rinsed in deionized water three times, oven-dried, and combusted in quartz tubes with cupric oxide wire at 900 °C for 3 hours to generate CO_2.

The marine shells analyzed in this study were manually cleaned in deionized water at the archaeological laboratories at UC Santa Barbara. Each shell was sectioned and a transect sample across the shells growth was taken with a dental drill. At Lawrence Livermore, carbonate samples (8–10 mg) were etched with 0.5N hydrochloric acid and rinsed with deionized water. These carbonate samples were placed in a 3 mL vacutainer and put under vacuum. After evacuation below 20 mtorr, these

samples were reacted with 0.5 mL phosphoric acid and hydrolyzed for 30–60 minutes at 90 °C to release CO_2.

The evolved carbon dioxide from shell and charcoal samples was reduced to graphite using a Cobalt powder catalyst and H_2 gas (Vogel et al. 1987). $^{14}C/^{13}C$ ratios were measured directly through AMS dating and ^{14}C ages were corrected using the conventions of Stuiver and Polach (1977). All ^{14}C dates are $^{13}C/^{12}C$ adjusted according to Stuiver and Polach (1977) to correct for mass-dependent fractionation. In this case, we assumed $\delta^{13}C$ values of −25 for charcoal and 0 for shell. ^{14}C ages were calibrated to calendar years using Calib. 4.0.2 (Stuiver and Reimer 1993). The local marine reservoir correction (ΔR) used to calibrate shell dates was 190 ± 40 (Stuiver and Braziunas 1993) and 24 years was subtracted from the charcoal dates prior to calibration as suggested by Stuiver et al. (1998) for terrestrial samples from the southern hemisphere.

RESULTS AND DISCUSSION

^{14}C ages for shell and charcoal pairs (samples 1–9) are listed in Table 1, along with calibrated ages before present (1 and 2 σ). The sampling locations of these samples are shown in Figure 1, and sample pairs are ordered in Table 1 according to stratigraphic position.

Based on the average global marine reservoir age (~400 years) and the ΔR for this region (190 ± 40), we expected the ^{14}C dates of shell (after $\delta^{13}C$ correction) to be roughly 600 years older than the charcoal dates. However, many of the paired shell and wood samples have essentially the same ^{14}C ages (Table 1). After calibrating all of the ^{14}C dates (using a ΔR of 190 ± 40), the charcoal samples were between 100 and 750 years older than the shell samples.

Table 1 CAMS dates are based on accelerator mass spectrometry. *C. concholepas = Concholepas concholepas (marine shell).* Calendar ages were determined using Stuiver and Reimer's (1993) Calib 4.0.2 and include the intercept (*denotes multiple intercepts) along with 1 and 2 σ age ranges. Shell dates were calibrated using the suggested ΔR value for the coast of Peru (190 ± 40) and 24 years was subtracted from all of the charcoal dates as suggested by Stuiver and Reimer (1998) for the southern hemisphere.

Lab nr (CAMS-)	Sample	Stratum	Level (cmbs)	Material dated	^{14}C age	Intercept	1 σ (BP)	2 σ (BP)	Difference
1770	1c	V	25	Charcoal	4500 ± 60	5114*	5294−4976	5313−4870	
1744	1s	V	25	*C. concholepas*	4950 ± 50	4986	5072−4877	5251−4831	−128
1770	9c	VII	40	Charcoal	4940 ± 60	5627	5711−5595	5842−5492	
1744	9s	VII	40	*C. concholepas*	4980 ± 60	5035	5219−4943	5284−4840	−592
1770	8c	VIII	53	Charcoal	4940 ± 60	5627	5711−5595	5842−5492	
1744	8s	VIII	53	*C. concholepas*	4990 ± 70	5042	5243−4945	5298−4836	−585
1770	7c	X	74	Charcoal	5020 ± 80	5276	5889−5615	5916−5592	
1744	7s	X	74	*C. concholepas*	4990 ± 50	5042	5221−4960	5279−4857	−684
1772	2c	XII	95	Charcoal	4930 ± 60	5634*	5661−5594	5838−5490	
1745	2s	XII	95	*C. concholepas*	4890 ± 60	4889	5023−4831	5199−4798	−745
1772	3c	XIII	115	Charcoal	4620 ± 60	5311	5446−5094	5468−5050	
1744	3s	XIII	115	*C. concholepas*	5050 ± 60	5213	5282−5034	5319−4936	−98
1783	6c	XVIII	150	Charcoal	5060 ± 80	5831*	5907−5659	5931−5602	
1744	6s	XVIII	150	*C. concholepas*	5070 ± 60	5240	5293−5048	5333−4960	−591
1770	4c	XXI	175	Charcoal	4940 ± 60	5627*	5711−5595	5842−5492	
1744	4s	XXI	175	*C. concholepas*	5080 ± 60	5251	5298−5060	5417−4968	−376
1770	5c	XXII	190	Charcoal	4530 ± 80	5131*	5310−4981	5449−4868	
1744	5s	XXII	190	*C. concholepas*	4950 ± 70	4986	5135−4864	5276−4818	−145

At a coarse level, shell and charcoal dates are somewhat comparable. However, there are some subtle differences that are potentially significant for understanding the nature of subsistence and settlement patterns regionally during this interval of time. The shell dates indicate that this midden accumulated rapidly between ~5300 and 4900 calendar years BP (<400 years and possibly even within the ^{14}C measurement uncertainty of 50–70 years), whereas the charcoal dates suggest a more gradual development between ~5800 and 5100 calendar years BP (~700 years). Three of the calibrated charcoal dates (paired samples 1, 3, 5) were relatively close to the shell dates and well within the confines of uncertainty inherent in ^{14}C dating. However, many of the charcoal samples (paired samples 2,4,6,7,8,9) were between 350 and 750 years older than the shell dates.
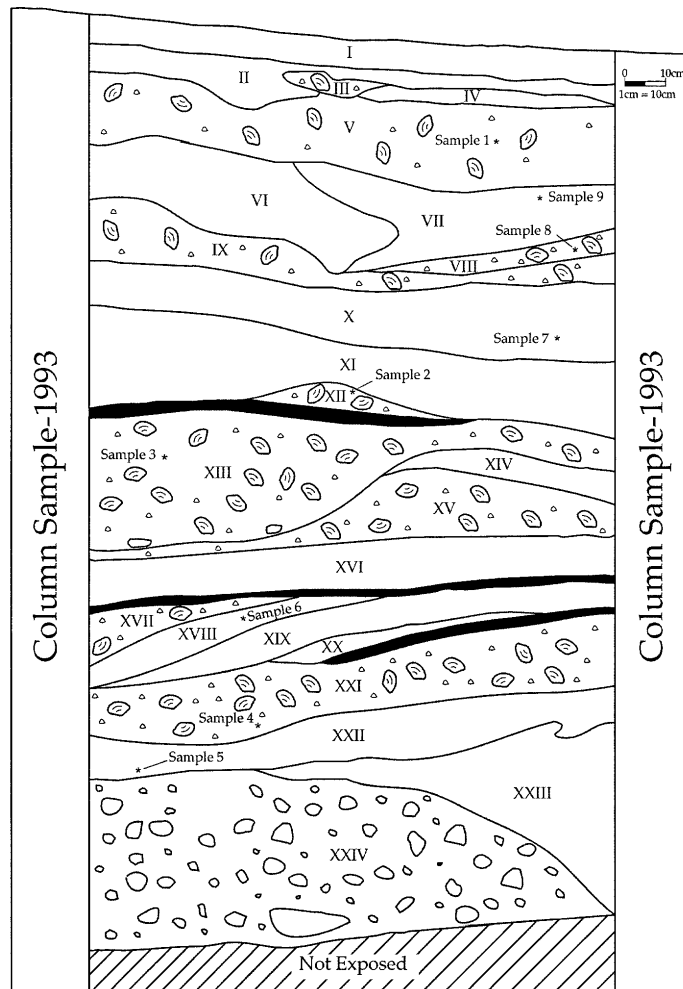

Figure 1 Stratigraphic section (railroad profile) indicating the position of shell and charcoal specimens that were collected for radiocarbon work

This was an unexpected result and the consistent nature of the pattern suggests a systematic underlying cause. One possible explanation for these discrepancies is that upwelling during the Middle Holocene was less intense and that the ΔR value for the region (190 ± 40) overcompensates for the marine reservoir effect for samples dating to this period. This could explain the smaller deviations visible in three of the pairs (samples 1,3,5), but cannot account for the charcoal ages 350–750 years older than the shell. Another, more likely possibility, is that the charcoal dates are older because people burned old wood at the site during the Middle Holocene. Today, the far south coast of Peru is a hyperarid environment and virtually no vegetation exists in the vicinity of the site, except at a remnant spring mouth at its northern edge where patches of grass are currently found. This spring was apparently more active during the Archaic Period, when it would have created a small desert oasis. Judging from other springs found along the coast in this area it might have supported grasses, reeds, and other plants, but likely few if any trees. Therefore, wood for house construction and fuel would have been limited. Driftwood was likely used and certainly could have been a source of old wood, a problem recognized in other regions (Erlandson et al. 1996). However, driftwood is limited on the beaches today due to the absence of extensively forested zones close to the coast. Alternatively, we suspect that wood was brought in from more forested interior areas, and it is likely that such wood was reused, possibly for generations, before it was finally burned as fuel.

The potential that prehistoric people along the Peruvian coast burned old wood introduces an unpredictable variable into the calibration process. Given this unpredictability, we argue that shell dates are more reliable in this particular context even with the uncertainties associated with the ΔR value for this region. This underscores the importance of obtaining better records of reservoir ages for this and other poorly studied coastal regions.

CONCLUSIONS

- [14]C dates on paired shell and charcoal samples from the railroad profile at Kilometer 4 were similar in many cases.
- This was unexpected because old carbon occurs in the marine reservoir.
- After calibration, most of the charcoal ages were 350–750 years older than the shell dates.
- We attribute these large disparities to the burning of old wood rather than instabilities in the marine reservoir.
- Potential problems associated with dating charcoal in coastal Peru highlight the importance of establishing more accurate ΔR values for this and other poorly studied coastal regions.

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