AMS ^{14}C dating of Holocene estuarine deposits: consequences of high energy and reworked foraminifera

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Abstract: Benthic foraminiferal species were used to AMS date Holocene sediments adjacent to transgressive overlap boundaries (TOB) in two high energy estuaries in North Spain. It had previously been recognised that the foraminifera could be divided into indigenous and exotic components. Whereas it was known that the exotic forms had been transported into the estuaries from the adjacent inner shelf, it was assumed that the indigenous forms were *in situ*. The AMS dates based on indigenous forms obtained from pairs of samples adjacent to the TOB showed an inverse relationship in three out of four boreholes (i.e., the age of the higher sample was older than that of the lower). The most probable explanation of this unexpected result is that there had been significant reworking of indigenous foraminifera especially associated with the transgressive episode. It is concluded that, in high energy estuaries, AMS dating on foraminiferal shells is influenced by transport and reworking even of the indigenous component. Therefore, if only foraminifera are available for dating, we recommend that closely spaced pairs of samples should be dated in order to assess the reliability of the results.

Key words: Benthic foraminifera, AMS radiocarbon dates, Holocene, trangressive overlap boundaries, estuaries, North Spain.

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Introduction

The north coast of Spain is a high energy coast that faces onto the Bay of Biscay. The continental shelf (0-200 m depth) is only 20 to 25 km wide in the studied region. Most of the coastline is erosional with extensive cliffs, and depositional areas with Holocene deposits are confined mostly to estuaries which have formed along lines of structural weakness such as faults and diapirs. The estuaries are small with a maximum length of around 15 km (Figure 1). The dominant wind direction and, therefore, the wave attack are from the north-west and these cause sand to be transported onto the eastern margin of the estuary mouths where it forms beaches and dunes. This process has operated for at least 6000 years (Cearreta, 1993).

It is only in the last few years that detailed studies have commenced on the postglacial history of development of the estuaries. Recent civil engineering work has involved drilling deep holes from which cores have been made available. Calcareous microfossils are well preserved in these sediments Because the ground waters are saturated in calcium carbonate (Gobierno Vasco, 1998). Furthermore we believe there was no contemporary dissolusion of foraminifera as the brackish waters were formed from a mixture of sea water and hard fresh water and were therefore not corrosive. We have used these well preserved foraminiferal assemblages to reconstruct past environments, ranging from inter/supratidal beach and dune to near-marine intertidal, and to determine the sequence of environmental changes (Cearreta and Murray, 1996; Cearreta, 1998). The low marsh assemblages represent depths of not more than 1 m below sea level. The non-marsh intertidal assemblages represent water depths of between 1 and around 4 m below sea level based on today's tidal range (1.2 m and 4.6 m for neap and spring tides respectively).

Of special interest are upward-shallowing sequences separated by a transgressive overlap boundary (TOB, Fletcher *et al*., 1993) which appear to record a rapid deepening e.g., from marsh to near-marine intertidal environments. In the Bilbao and Santoña estuaries (Figure 1) a TOB has been identified (Cearreta and Murray, 1996; Cearreta, 1998). In the absence of any ^{14}C dates for these estuaries it was only by comparison with published results from other parts of the Bay of Biscay that an age interpretation of the TOB could be made (3000 yrs BP).

The aims of this study were to obtain ^{14}C dates of the TOBs in Santoña and Bilbao estuaries and also to place these results into the regional framework of sea-level changes in the Bay of Biscay. Benthic foraminifera were used for dating because they are generally abundant, whereas macrofaunal groups, such as molluscs, are rarely encountered in the cores. The results provide new insights on the problems of obtaining reliable dates from high energy environments. .

Materials and methods

Boreholes were drilled using a rotary drill that produced a core approximately 10 cms in diameter. In order to avoid downhole contamination, samples were taken just from the central part of the core. The Bilbao cores were drilled in 1988-1990 and sampled in March 1991, and the Santoña cores were drilled and sampled in 1993. In the laboratory, the samples were dried in an oven at 120°C and then weighed. The target weight for analysis was 300 g. They were wet sieved through a 63 µm mesh, dried, and weighed again to determine the proportion of sand. The foraminifera were concentrated using trichloroethylene, dried and then stored in a glass container.

The preserved typical marsh foraminifera were not used for dating because they are not calcareous. Eight samples with calcareous foraminifera from four selected boreholes in the two estuaries were dated using the AMS ^{14}C method. For each core two samples were chosen, one each from either side of the previously recognised TOB (Figure 1). In the study of the recent foraminiferal fauna of the Santoña estuary those species found living today were regarded as indigenous and those found only as dead tests were considered to be exotic forms transported in from the adjacent high energy shelf (Cearreta, 1988). In the study of different boreholes in the Santoña and Bilbao estuaries these two groups were distinguished throughout the Holocene record (Cearreta and Murray, 1996; Cearreta, 1998). The target weight of sample for AMS dating was 20 mg. Where possible, we picked only one indigenous species for dating (*Ammonia beccarii*) but in four samples it was necessary to use a combination of several species (*A. beccarii*, *Elphidium williamsoni*, and *Haynesina germanica*). In three of the analysed samples it was still not possible to obtain 20 mg of tests as the indigenous assemblage formed only a small proportion of the whole assemblage. Samples were prepared to CO_2 and/or graphite at the NERC Radiocarbon Laboratory, East Kilbride, Scotland, and then sent to AMS laboratories for ^{14}C analysis. Samples were hydrolysed to CO_2 using 100% ortho-phosphoric acid at 25°C. Samples with publication codes OXA/ were analysed as CO_2, (as they were too small for graphitising) by AMS at the Oxford Radiocarbon Accelerator Unit, University of Oxford, England (Table 1). The remaining samples were converted to graphite by Fe/Zn reduction (Slota *et al*., 1987) and sent to the NSF-AMS Facility, University of Arizona, USA, for analysis. The results are reported as conventional radiocarbon age BP (before A.D. 1950). Overall analytical errors are quoted as 1σ. No correction has been made for reservoir age.

Results

AMS results are listed in Table 1 and details of individual boreholes are given below. In Table 1 the sample depth is given in metres downcore below modern sea level.

The SCA borehole (Santoña estuary) was drilled in the upper estuary (43º23'10"N, 3º27'36"W; 2.4 m above mean sea level). The environment at sample SCA-39, 0.15 m below the TOB (situated at -17.5 m), was interpreted as a low marsh with an indigenous assemblage of *Jadammina macrescens* 44% and *Miliammina fusca* 25%, with 21% exotic tests, and a Fisher alpha diversity of 4.5 (Fisher *et al*. 1943). That of sample SCA-38, 0.15 m above the TOB, was interpreted as near-marine intertidal because the indigenous assemblage is dominated by *A. beccarii* 10% and *H. germanica* 9%, with 66% exotic tests, and a Fisher alpha diversity of 5 (Cearreta and Murray, 1996). The lower sample (SCA-39) was dated at 6260 ± 105 radiocarbon yrs BP, and the upper sample (SCA-38) at 6715 ± 110 radiocarbon yrs BP.

The SST borehole (Santoña estuary) was drilled in the upper estuary (43º26'54"N, 3º28'20"W; 2.8 m above mean sea level). The environment at sample SST-37, 0.15 m below the TOB (situated at -15.6 m), was interpreted as a low marsh with an indigenous assemblage of *H. germanica* 25%, *A. beccarii* 19% and *J. macrescens* 8%, with 39% exotic tests, and a Fisher alpha diversity of 8.5. The environment at sample SST-36, 0.15 m above the TOB, was interpreted as near-marine intertidal because the indigenous assemblage is dominated by *A. beccarii* 10%, with 81% exotic tests, and a Fisher alpha diversity of 8 (Cearreta and Murray, 1996). The lower sample (SST-37) was dated at 1395 ± 45 radiocarbon yrs BP, and the upper sample (SST-36) at 3900 ± 150 radiocarbon yrs BP.

The SR8 borehole (Bilbao estuary) was drilled in the lower-middle estuary (43º18'30"N, 2º58'26"W; 3.7 m above mean sea level). The environment at sample SR8-14, 0.25 m below the TOB (situated at -7.55 m), was interpreted as brackish intertidal because of an indigenous assemblage of *A. beccarii* 49%, *H. germanica* 24% and *E. oceanensis* 14% with a Fisher alpha diversity of 3.5. The environment at sample SR8-13, 0.25 m above the TOB, was interpreted as near-marine intertidal with an indigenous assemblage of *A. beccarii* 38% and *H. germanica* 19% and Fisher alpha diversity of 6. The proportions of exotic tests are 9% and 36% respectively (Cearreta, 1998). The lower sample (SR8-14) was dated at 7545 ± 65 radiocarbon yrs BP, and the upper sample (SR8-13) at 6515 ± 65 radiocarbon yrs BP.

The S15 borehole (Bilbao estuary) was drilled in the lower estuarine area (43°19'28"N, 2°59'46"W; 4.9 m above mean sea level). The environment at sample S15-28, 0.25 m below the TOB (situated at -11.85 m), was interpreted as brackish intertidal with an indigenous assemblage of *A. beccarii* 40% and *H. germanica* 12%, with 30% exotic tests, and a Fisher alpha diversity of 5. The environment at sample S15-26, 0.75 m above the TOB, was interpreted as near-marine intertidal because the indigenous assemblage is dominated by *A. beccarii* 27% and *H. germanica* 9%, with 51% exotic tests, and a Fisher alpha diversity of 6.5 (Cearreta, 1998). Although representing the same environment, sample S15-27 located at 0.25 m above the TOB did not contain enough indigenous tests for dating. The lower sample (S15-28) was dated at 7765 ± 65 radiocarbon yrs BP, and the upper sample (S15-26) at 8005 ± 65 radiocarbon yrs BP.

Discussion and conclusions

The results presented here are for pairs of samples, one taken below and one above the TOB in four boreholes. In one core (SR8 from the Bilbao estuary), the upper sample is younger than the lower. However, in the other three cores, the upper sample is older than the lower even when the error bands are taken into account. This is clearly unexpected and requires an explanation. Possible explanations are: contamination of the samples, reservoir age affecting the geochemistry of the foraminiferal calcite, and reworking of foraminiferal tests into the assemblages.

Contamination of the samples

Because of the rotary drilling process used to collect the cores, the only possibility of contamination as the drilling progresses downwards could be of younger material from above. This means that in case of contamination samples should be dated younger than their real age. However, as pointed out in the Methods section we sampled only the central part of the cores in order to avoid this problem.

Reservoir age and ^{14}C dates

There is no known published information on the apparent age (reservoir effect due to slow mixing of surface and deep ocean waters) of marine or brackish waters in the study area and no correction has therefore been made for reservoir effects. The apparent age of the surface waters of the North Atlantic is around 400 years (Bard *et al*., 1987) and contemporary marine molluscs for coastal waters of the UK have been found to have an apparent age of 405 ± 40 yrs (Harkness, 1983). Around Denmark, the reservoir age of the open waters falls in the range

290-500 years (weighted average 377 ± 16 yrs) but for lagoon waters it ranges from 470-900 years (Heier-Nielsen, Heinemeier *et al*., 1995). They considered that the lagoon samples had a mixture of marine North Sea water and freshwater containing old carbonate derived from the surrounding land. Thus, in this case the measured ages would be older than the true ages. There are widespread carbonate rocks of Cretaceous age along the North Spain coast so fresh waters flowing into the estuaries are saturated in dissolved old carbonate (hard-water effect) (Gobierno Vasco, 1998). Thus, it is possible that throughout the Holocene the brackish waters here have a higher reservoir age than the open marine waters. At each TOB, where there is a change from brackish below to more marine above, the calculated dates below could be older than they should be. However, the results show the opposite effect.

Influence of reworking on measured radiocarbon dates

In the original studies (Cearreta and Murray, 1996; Cearreta, 1998), the assemblages were divided into indigenous and exotic components and the interpretation of the environments was based only on the indigenous fauna. The exotic components are dominated by epifaunal taxa (especially *Cibicides lobatulus* and *Rosalina* spp.) which live attachted to rocks, hydroids, holdfasts of algae, mollusc shells, and other firm substrates on the inner shelf. They are known to be exotic as they are not found living in the estuaries and also such substrates are rarely present in these environments. Because the inner shelf is a high energy environment, after death the epifaunal tests are liable to be detached from the substrate and they are then available to be transported into the estuaries. Although they are most abundant in the lower estuary, exotic tests are also present in the upper estuary. The indigenous components comprise mainly infaunal species which are found living in the estuaries. Although it might be assumed that dead indigenous tests are preserved *in situ*, they are potentially likely to be transported by tidal currents within the estuarine environment so that although they are indigenous they are not *in situ*. Furthermore, when indigenous tests are reworked from older Holocene deposits into contemporary sediments having the same faunal assemblages, this is virtually impossible to recognise.

The most comprehensive study of the effects of reworking of older tests into contemporary environments is that of Heier-Nielsen, Conradsen *et al*. (1995) who studied the Skagen spit in northernmost Denmark. They carried out dating on mollusc shells and compared these with those obtained on foraminifera from the same samples. The molluscs gave an age curve which was smooth and there was a progression of ages downcore. It was concluded that the molluscs were *in situ* and that the age curve was reliable. By contrast, the foraminiferal record gave different dates, with some reversals, and only in a few cases did the two age curves coincide. The most reliable foraminifera were infaunal taxa presumed to be *in situ*. Others were interpreted as being reworked older forms (for example epifaunal miliolids). These introduced age errors as great as 5000 years.

Each TOB represents an increase in the amount of energy because it marks a change from upper intertidal zone to lower intertidal zone and therefore greater exposure to water movement. Both the proportion of sand and of exotic tests could be regarded as measures of the amount of transport and reworking of sediment. In order to demonstrate this, we have calculated the mean values for the environmental unit both above and below the TOB. With one exception (SST) there is an upward increase in the mean sand content across the TOB (SCA 46 to 89%, SR8 10 to 84%, S15 9 to 59%) and even in SST, where all the sediments are sandy, the decrease in mean sand content above the TOB is not very great (75 to 62%). However, in all cases there is an upward increase in mean relative abundance of exotic tests (SCA 10 to 69%, SST 47 to 80%, SR8 17 to 59%, S15 30 to 76%). A greater error might therefore be expected from the boreholes taken close to the estuary mouth compared with those taken in the upper part of the estuary. This conclusion is supported by the present study. In the upper part of the Santoña estuary (core SCA) material from below the TOB was dated 6260±210 radiocarbon yrs BP and 6715±220 from above, whereas for core SST near the mouth the values were 1395±45 and 3900±300 respectively. In the Bilbao estuary, for core SR8 from the middle estuary the date below the TOB is older than that above as would be expected (7545±65 and 6515±65 respectively). However, core S15 from near the mouth was dated 7765±65 from below the TOB and 8005±65 from above.

Consequences of reworking for dating sediments in high energy estuaries

Foraminifera are the most abundant calcareous fossils in the Holocene successions of estuaries on the north coast of Spain. Although we differentiated between indigenous and exotic components of the assemblages, it has proved to be a false assumption that the indigenous forms are all contemporary and buried *in situ*. The most likely explanation of the inverted dates recorded at the TOBs is that the samples above the TOB include a significant content of older reworked indigenous tests. This is an attribute of a high energy environment and it accords with similar observations made in northern Denmark (Heier-Nielsen, Conradsen, et al. 1995). Thus, none of the dates can be considered as reliable and therefore the aims of the original study could not be achieved.

However, the results provide new insights into the problem of obtaining reliable AMS dates from high energy environments. First, reworking of older tests into indigenous assemblages is an important process in high energy estuaries. Second, the consequence of this is that the measured AMS dates are not reliable. It must be emphasised that it is only because closely spaced pairs of samples were dated in this study that the problem of unreliability was identified. It follows from this that single, isolated dates obtained from foraminiferal tests in cores from high energy estuaries cannot be tested for reliability and should therefore be treated with caution.

In those cases where foraminifera are the only calcareous shells available for dating Holocene successions, we recommend that closely spaced pairs of samples should be dated in order to assess the reliability of the results. We would expect the results to be unreliable in medium to high energy environments. Even in low energy estuaries, channel migration may cause erosion of older deposits leading to the introduction of reworked foraminiferal tests, so the problem of unreliability may affect any type of estuary. Without such checks on pairs of samples, the validity of isolated dates is questionable.

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Caption to the Figure

Figure 1 Geographical location of the estuaries studied (1 = Santoña, 2 = Bilbao) and summary of the four boreholes from which samples have been dated. For each borehole the sequence of environments and the Trangressive Overlap Boundaries (TOB) are given together with the interpreted environments of deposition based on the indigenous foraminiferal component. The right hand column gives details of the exotic component. The positions of the dated samples are given to the left of each borehole. For details see Table 1.

Table 1 AMS radiocarbon dates.

Sample	Publication Code	Depth (m)	Material	Radiocarbon age BP (1σ)
SCA-38	OXA/W671-15	-17.1	various sp.	6715±110
SCA-39	OXA/W671-16	-17.4	various sp.	6260±105
SST-36	OXA/W671-14	-15.45	various sp.	3900±150
SST-37	AA-26385	-15.75	various sp.	1395±45
SR8-13	AA-26382	-7.3	*A. beccarii*	6515±65
SR8-14	AA-26386	-7.8	*A.beccarii*	7545±65
S15-26	AA-26383	-11.1	*A. beccarii*	8005±65
S15-28	AA-26384	-12.1	*A. beccarii*	7765±65