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Seasonality of δ^{13}C and C/N ratios in modern and mid-Holocene sediments in the Severn Estuary Levels, SW Britain

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Abstract: Bulk organic δ^{13}C and C/N ratios from mid-Holocene salt-marsh deposits with sedimentary banding reveal subtle but significant differences between coarse- and fine-grained deposits. These are consistent with findings from seasonally sampled modern silts, and with the interpretation, on physical and palynological grounds, of the fine-grained and coarse-grained components as warm-season and cold-season deposits, respectively. The control is considered to be seasonal variations in the character of the organic matter supplied.

Key words: Severn Estuary, Holocene estuarine silts, δ^{13}C and C/N values, seasonal effects.

Introduction

In the Severn Estuary Levels (Figure 1), a distinctive facies of Holocene salt-marsh silts that display seasonality through the presence of sedimentary banding is common (Allen, 1990, 2004; Allen and Haslett, 2002, 2006, 2007), and occurs elsewhere in European Holocene deposits (eg, Tessier, 1998). So far, the seasonal signal has been demonstrated on independent textural and palynological grounds (Allen, 2004; Dark and Allen, 2005), but not geochemically.

Here we draw attention to the potential of bulk δ^{13}C and C/N ratios for the further independent detection of seasonality in this facies. Hitherto, in relation to Holocene salt-marsh deposits, isotopes have been applied to issues of environment and sea-level change (eg, Wilson *et al*., 2005a,b). Palaeoenvironmental studies of seasonal changes in source inputs have been limited, largely because of the apparent scarcity of seasonally defined sediments. There has, however, been a rapid expansion of their application to the organic inputs of modern estuaries (Lamb *et al*., 2006). Chiefly in connection with carbon cycling, several workers examined seasonal variations in the δ^{13}C and C/N of organic supplies to contemporary estuaries (eg, Cifuentes *et al*., 1988; Ogawa and Ogura, 1997; Kaldy *et al*., 2005). Weiguo *et al*. (2003) showed that the δ^{13}C of fluvial particulate organic matter (POC) is highly responsive to seasonal changes in the proportions of C_3 and C_4 catchment vegetation, and this has the potential to be preserved in sediments.

We describe below the carbon isotopic composition of plant tissues present in modern sediments from the Severn Estuary, collected over a year from a salt marsh and mudflat preparatory to broader and more intensive studies (Dark and Allen, 2006), and compare it with mid-Holocene banded silts of salt marsh origin. The Severn Estuary – a large, complex and energetic system (Dark and Allen, 2005) – displays seasonal variations in the relative importance of different sources of plant-related and mineral debris, which are expected to be registered in the deposited muds. The input of mineral and variously degraded plant tissues (exogenous sources) increases from the warm to the cold season. As well as affording root matter, the plants that flourish on the marshes during warm months decay to produce litter in the cold season (endogeneous sources), some finding its way during storms into tidal waters. The estuarine water-body is itself a huge reservoir of detritus of all origins.

That seasonality should be sought in estuarine sediments is important for several reasons. Sedimentologically, it advances understanding of the role and pace of sea-level and coastal change during Holocene sequence-building (Allen and Haslett, 2002, 2006). Environmentally, it is indicative of climate and climate-variability (Allen and Haslett, 2006). Archaeologically, it can provide short-range, high-resolution chronologies and, especially through work on footprints (Allen *et al*.,

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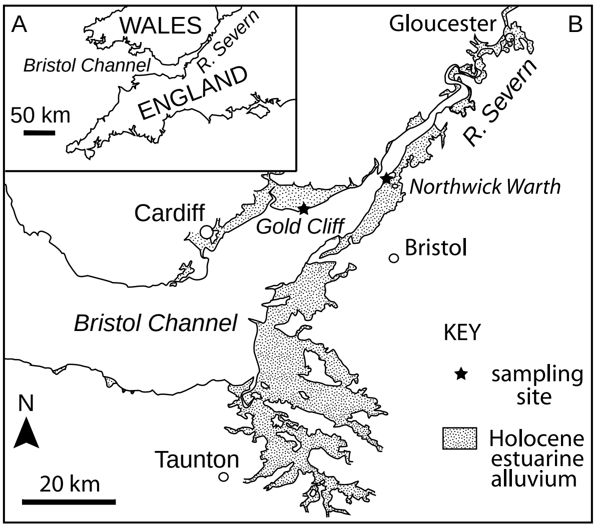

Figure 1 Southwest England (A) with the Severn Estuary Levels (B) showing the localities and sample stations mentioned in the text

2003), help identify the seasons when wild herbivores and humans exploited coastal wetlands.

Sites and field sampling

Northwick and Aust Warths

Modern sediment samples and plant material were collected from the mudflat known as Northwick Oaze and the formerly grazed salt marsh called Aust Warth, the northeastward extension of Northwick Warth (Figure 1B, 2). Active marshes here occur at three, seaward-descending, levels (Allen and Rae, 1987; Strawbridge *et al.*, 2000; Haslett, 2006), indicative at progressively later dates of inception after a phase of downcutting and marsh-edge retreat (Allen, 1989).

The marsh vegetation (Smith, 1979; Dark and Allen, 2006) is tidally zoned (Figure 2). Uppermost is *Elytrigia atherica* (sea couch, *Agropyron pungens*; 7.7–8.2 m OD), sharply transitional to *Festuca rubra* (red fescue; *c.* 7.2–7.7 m OD), in turn grading rapidly to *Puccinellia maritima* (common salt-marsh grass; *c.* 6.2–7.2 m OD). *Spartina anglica* (common cord-grass) gradually increases seaward downward from an altitude of 7.4 m OD, dominating from 4.2–5.9 m OD together with *Salicornia* (glasswort). Five plant species (above-ground biomass) were sampled for geochemical analysis.

Silt suspended in the estuarine waters was trapped at three stations over periods of two days on three occasions in 2004 when sources of plant material were likely to differ (early January, early May, latest August). Anchored, shallow plastic trays and large, partly buried plastic beakers were positioned at two points (stations 2 and 3) on Aust Warth and at a site (station 1) on Northwick Oaze (Figure 2). The beakers were not used at stations 2 and 3, however, until May and August. Tens to hundreds of grams of mud accumulated in a trap on each occasion, nominally representing four tidal cycles. Grain-size distributions were measured with a Coulter LS230 laser-granulometer, following a described protocol (Allen, 2004) that excludes hydrogen peroxide treatment (Allen and Thornley, 2003). The sediments proved to be sandy-clayey silts. In terms of the average arithmetic mean size, the May and August deposits were 15% and 29% finer-grained, respectively, than the January ones. As the tray and beaker samples did not differ significantly, isotope analyses were limited to the organic fraction present in the former, as described below.

Gold Cliff

The mid-Holocene deposits exposed on the Welsh coast of the Severn Estuary (Figure 1) include two, metre-scale, units of banded estuarine silt of salt-marsh origin traceable laterally for *c.* 15 km (Allen and Haslett, 2006). A short monolith from the lower bed, dated to the middle of the second half of the fifth millennium BC, was taken just to the east of Gold Cliff and subsampled in contiguous 5 mm slices followed by laser analysis (Allen, 2004). Values for the arithmetic mean grain-size of these sandy-clayey to clayey-sandy silts display through a sequence of five complete bands a bold pattern of sharp maxima (subsamples W_{1-5}) and minima (subsamples S_{1-5}) considered to be seasonally determined (Figure 3). Independently, these subsamples proved to have pollen and spore assemblages that differed subtly but significantly in terms of season of deposition (Dark and Allen, 2005). Isotope and C/N analyses were made of the organic fraction extracted from these subsamples.

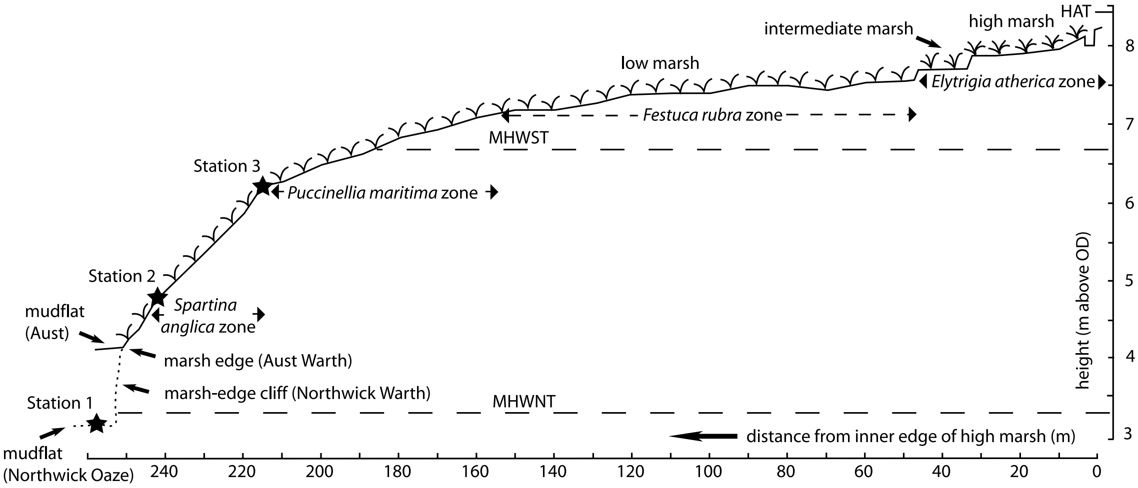

Figure 2 Transect across Aust Warth showing the three levels of salt marsh, the botanical zonation and the location of marsh and mudflat sampling stations (after Dark and Allen, 2006). The marsh-edge cliff at Northwick Warth and station 1 on the adjoining mudflat (Northwick Oaze) are superimposed schematically on the transect. HAT, highest astronomical tide; MHWST, mean high-water of spring tides; MHWNT, mean high-water of neap tides

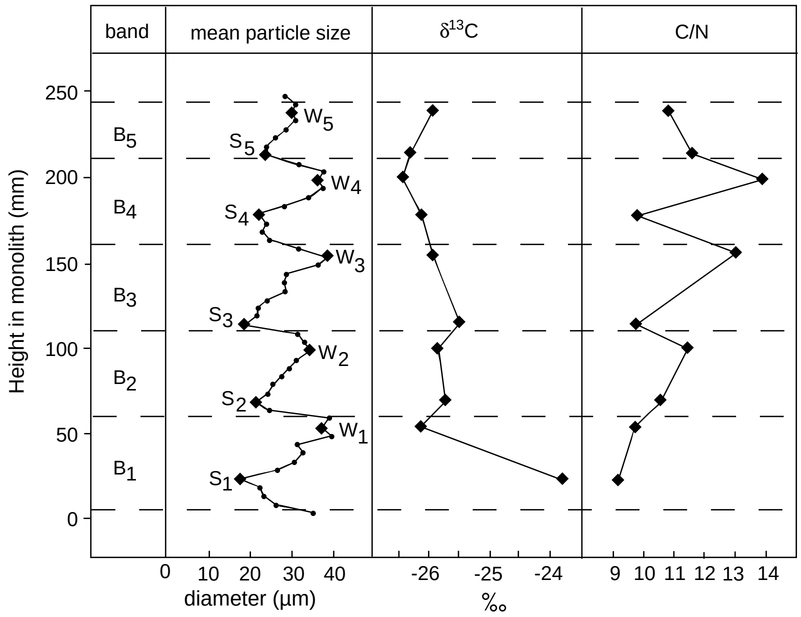

Figure 3 Summary of textural data (data of Allen, 2004; Dark and Allen, 2005) and geochemical analyses (this paper) for the mid-Holocene banded silts collected from Gold Cliff. The sedimentary bands B_1–B_5 are as defined by Dark and Allen (2005)

C/N and δ^{13}C isotopic methods

Plant specimens from Aust Warth were dried after collection and freezer-milled to a fine powder. The suspended sediments (Northwick and Aust Warths) and mid-Holocene silts (Gold Cliff) were freeze-dried, ground to a fine powder and treated with 5% HCl overnight to remove inorganic carbon (shell debris, early-diagenetic carbonate).

^{13}C/^{12}C analyses were performed on plant and sediment powders by combustion in a Carlo Erba 1500 online to VG Triple Trap and Optima dual-inlet mass spectrometer with δ^{13}C values calculated to the VPDB scale using a within-run laboratory standard calibrated against NBS-19 and NBS-22. Replicate analysis of well-mixed samples indicated a precision of $\pm <0.1$‰ (1σ). The weight ratios of organic carbon to total nitrogen (C/N) are analysed on the same instrument and calibrated against an acedanalid standard. A precision of ±0.1 (1σ) was indicated by replicate analyses.

C/N and δ^{13}C isotope results and interpretation

The application of δ^{13}C and C/N ratios to estuarine sediments rests on the observation that such sediments include organic material both from indigenous plants and material transported with the tide or from a river catchment.

Tidal salt marshes typically are densely covered with vascular plants, whereas mudflats and sandflats found at lower levels have only a variable coating of algae and tidal-derived organic material (mostly phytoplankton). As the latter normally has higher δ^{13}C values than freshwater phytoplankton and indigenous vascular plants, height within the tidal frame can often be deduced from sediment δ^{13}C values and C/N ratios (eg, Wilson *et al.*, 2005a,b). C_3 vascular plants typically have δ^{13}C values of between −32‰ and −21‰ (Deines, 1980) and relatively high C/N ratios of >12 (Prahl *et al.*, 1980), as they consist chiefly of N-poor lignin and cellulose. In contrast, C_4 plants (Deines, 1980) have much higher δ^{13}C ratios of around −13‰ (range −17‰ to −9‰). On British salt marshes, C_4 plants have only become widespread since the nineteenth century (eg, Preston *et al.*, 2002) and their remains will be sparse or absent from pre-modern deposits. Freshwater algae in C_3-dominated environments tend to have lower δ^{13}C values (−26‰ to −30‰) (Schidlowski *et al.*, 1983; Meyers, 1994) than marine algae (−16‰ to −23‰) (Haines, 1976; Tyson, 1995). Again in C_3 environments, fluvial $\delta^{13}C_{POC}$ values reflect the relative contributions of freshwater phytoplankton (−30‰ to −25‰) and particulate terrestrial organic matter (−33‰ to −25‰) (eg, Salomons and Mook, 1981; Middelburg and Nieuwenhuize, 1998; Barth *et al.*, 1998).

Aust Warth and Northwick Oaze

The limited range of plant species available for analysis is typical of western British marshes (Figure 2, Table 1). *Elytrigia altherica* (high marsh) has a δ^{13}C value of −27.1‰, whereas the lower marsh plants yield a mean value of −26.6±0.3‰, excepting *Spartina anglica* (−12.4‰). The latter, a C_4 plant, arose *c.* 1890 as an amphidiploid from *S.* x *townsendii* (itself a hybrid of the native *S. maritima* and introduced *S. alterniflora*) (Goodman *et al.*, 1969). Both *S. anglica* and *S.* x *townsendii* are invasive and widely planted.

Turning to the suspended silts (Figure 3, Table 1), some geochemical distinctions are possible between the seasons, albeit small in terms of δ^{13}C. Overall, the January samples are different from the quite similar muds collected in May and August. The mean δ^{13}C from the three locations is marginally lower in January (−24.1±0.3‰) than in May and August (−23.9±0.1‰, −23.8±0.0‰), although the difference is almost analytical error. C/N ratios are higher in January (17.2±1.4) compared with May (14.4±1.6) and August (13.0±0.6). Higher C/N ratios (16–19) suggest an increased proportion of river-imported detritus (C_3 plants), an interpretation supported by the slightly lower δ^{13}C values (−24.1‰) than in summer (−23.8‰). Slightly higher δ^{13}C (−23.8‰) and lower C/N (12–15) suggest proportionately more algae in the summer months and less organic material from fluvial/terrestrial sources. British intertidal phytoplankton typically have high δ^{13}C ratios (−16‰ to −21‰) and low C/N ratios (4–6), and so summer phytoplankton blooms would act to increase the contrast with the material imported by rivers in winter. There seems to be little discernible difference in δ^{13}C and C/N between the mudflat (station 1) and the marsh (stations 2, 3), suggesting that the patterns we describe are consistent across the area.

Table 1 Data for $\delta^{13}C$ and C/N on organic material from Aust Warth and Northwick Oaze and from Gold Cliff, Severn Estuary Levels

Sample description	Environment	$\delta^{13}C_{organic}$ ‰	C/N
Modern plant species			
Elytrigia atherica	high marsh	−27.1	110.4
Puccinellia maritima	low marsh	−26.3	27.8
Aster tripolium	low marsh	−26.7	15.1
Triglochin maritima	low marsh	−26.9	23.2
Spartina anglica	very low marsh	−12.4	27.6
Mean (±1σ)		−23.9±6.4	40.8±39.3
Modern suspended organic matter (January)			
Northwick Oaze	mudflat (st. 1)	−23.9	18.7
Aust Warth	salt marsh (st. 2)	−24.0	16.0
Aust Warth	salt marsh (st. 3)	−24.4	16.8
January mean		−24.1±0.5	17.2±1.4
Modern suspended organic matter (May)			
Northwick Oaze	mudflat (st. 1)	−23.9	13.3
Aust Warth	salt marsh (st. 2)	−24.0	13.8
Aust Warth	salt marsh (st. 3)	−23.9	16.3
May mean		−23.9±0.1	14.4±1.6
Modern suspended organic matter (August)			
Northwick Oaze	mudflat (st. 1)	−23.8	13.7
Aust Warth	salt marsh (st. 2)	−23.8	12.5
Aust Warth	salt marsh (st. 3)	−23.8	12.9
August mean		−23.8±0.0	13.0±0.6
Mid-Holocene banded silts (Gold Cliff)			
S_1	salt marsh	−23.9	9.2
S_2	salt marsh	−25.8	10.6
S_3	salt marsh	−25.6	9.8
S_4	salt marsh	−26.2	10.0
S_5	salt marsh	−26.4	11.8
Mean		−25.6±1.0	10.3±1.0
W_1	salt marsh	−26.2	9.8
W_2	salt marsh	−25.9	11.6
W_3	salt marsh	−26.0	13.2
W_4	salt marsh	−26.5	14.1
W_5	salt marsh	−26.0	11.0
Mean		−26.1±0.2	11.9±1.7

Gold Cliff

The mid-Holocene data from Gold Cliff (W_{1-5}, S_{1-5}) exhibit cyclical patterns (Figures 3 and 4; Table 1) that can be tentatively linked to the above observations from the modern environment, though generally the proportions of organic C and N are very low (< 1%), and thus the C/N values should be treated with some caution.

Overall, the S subsamples yield lower C/N ratios (*c*. 10) and slightly higher $\delta^{13}C$ values (mean −25.6±1.0‰) than the W subsamples, suggesting perhaps a higher algal content and relatively lower fluvial input. A greater river supply is indicated by the W subsamples, which yield higher C/N ratios (*c*. 12) and slightly lower $\delta^{13}C$ values (mean −26.1±0.2‰), although again the $\delta^{13}C$ difference is close to analytical error. The cyclical pattern is especially clear in the case of bands B_2, B_3 and B_4, but for B_1 and B_5 is less marked, pointing in their case to possibly little seasonal difference between the sources of plant matter. Standing out from all the others is subsample S_1, with its higher organic content, much higher $\delta^{13}C$ ratio and lowest C/N value, which may record a particularly fruitful or prolonged growing season (see below).

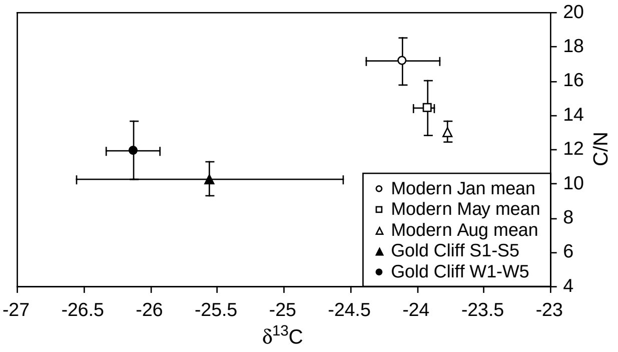

Figure 4 Mean $\delta^{13}C$ values versus mean C/N ratios for modern samples collected from Aust Warth and Northwick Oaze in January, May and August, and the mid-Holocene subsamples W_{1-5} and S_{1-5} from Gold Cliff. Error bars represent one standard deviation. See also Table 1

Discussion

The January silts from Aust Warth and Northwick Oaze are significantly coarser grained than the May and August deposits because, as argued elsewhere (Allen, 1990, 2004), winter waters are observed to be colder and stormier than in other seasons, and therefore able to hold coarser sediment. By the same token, the W and S subsamples from Gold Cliff are respectively 'cold-season' and 'warm-season' deposits (Allen, 2004). These sets of subsamples also differ palynologically, in a manner consistent with modern patterns of flowering and sporulation (Dark and Allen, 2005), affording independent proof of the same seasonality. Geochemically, we suggest that the modern silts also exhibit seasonal differences that, used as a model, assign the S and W subsamples from

Gold Cliff as otherwise to warm and cold seasons, respectively (Figure 4, Table 1).

There are nonetheless differences in the ranges of the $\delta^{13}C$ and C/N values between the modern and mid-Holocene sediments, with the modern ones having higher $\delta^{13}C$ values ($-24.4‰$ to $-23.8‰$) and C/N ratios (12.5–18.7) than the earlier silts ($-26.5‰$ to $-23.9‰$; 9.2–14.1). Two factors may explain this.

The presence of *Spartina anglica* may account for the higher values in the modern silts. C_4 grasses can have C/N ratios of above 30 and significantly higher $\delta^{13}C$ ratios than C_3 plants (Meyers, 1994), which would serve to increase the bulk sediment C/N ratios and $\delta^{13}C$ values of the modern samples compared with the probably C_4-free older ones. The modern and older marshes could have differed botanically in other ways, as suggested by the much higher proportion of Chenopodiaceae pollen in the mid Holocene than the modern samples (Dark and Allen, 2005, 2006).

A second possible factor is the abundant, mining-related, detrital coal-dust present in the modern sediments of the Severn Estuary, which can be removed or quantified using various techniques (Allen, 1987; French, 1993, 1998). However, as dissolved organic carbon can be adsorbed by detrital coal, the physical removal of coal from samples could bias the biogeochemistry of what remained (Hedges and Keil, 1995; Ransom *et al.*, 1997). Removal was not attempted, but we analysed steam coal from the region and detrital coal from silts of mid twentieth-century date (effectively the average coal in the system). These gave mean values for $\delta^{13}C$ and C/N of $-23.3 \pm 0.9‰$ and 41.0 ± 0.3, respectively. If the $\delta^{13}C$ values and C/N ratios of detrital coal were significantly different from river-derived organic matter, then this addition may be a significant contaminant to sedimentary organic material. As Table 1 shows, however, the two ratios fall squarely in the ranges for the salt-marsh plants. As increases in the amount of coal in the silts will correspond to growth in the relative proportion of river-imported terrestrial plants compared with halophytes (with higher $\delta^{13}C$ values and lower C/N ratios), the effect of detrital coal on $\delta^{13}C$ and C/N can safely be neglected.

As commonly observed elsewhere (Lamb *et al.*, 2006), the $\delta^{13}C$ values for the mid-Holocene silts are lower than the otherwise similar modern sediments. Diagenetic changes in bulk sediment organic $\delta^{13}C$ can be particularly significant, and take place relatively quickly because of the differing isotopic compositions of labile and refractory plant compounds (eg, Ember *et al.*, 1987; Chmura and Aharon, 1995; Mallamud-Roam and Ingram, 2001). The refractory lignin of vascular plants, for example, is lower in $\delta^{13}C$ by 2–6‰ relative to the whole organism. Thus $\delta^{13}C$ values are forced toward those of lignin by the preferential decay of labile compounds (Ember *et al.*, 1987; Benner *et al.*, 1987, 1991; Wilson *et al.*, 2005b).

The correspondence of the $\delta^{13}C$ and C/N values to independent textural and pollen-based interpretations of the banded mid-Holocene salt-marsh silts from Gold Cliff suggests that the geochemical data also record seasonal changes in organic-matter sources (Figures 3 and 4; Table 1). In the coarse-grained sediments (W_{1-5}) there are lower total pollen concentrations, pollen from chiefly autum or early-spring flowering plants, and relatively high proportions of pollen and spores likely to have been transported by rivers during the cold season when discharges are greatest. The fine-grained parts (S_{1-5}) have higher total pollen concentrations and higher proportions of late-spring to summer flowering plants, pointing to origin in the warmer months. The interpretation of the isotope and C/N data supports these various observations. For instance, the degree of fluvial transport, apparently greatest during the cold months, is reflected in the higher C/N values (*c.* 12) and marginally lower $\delta^{13}C$ values (mean $-26.1‰$) for the W silts than the S deposits. Additionally, the larger contrast in mean particle size between S_1 and W_1 in band B_1 is also evident in the $\delta^{13}C$ ratios, with S_1 yielding a particularly high value compared with other warm-season deposits. This could reflect an unusually strong algal bloom on the marsh that would also explain the low C/N ratio of 9.2. Similarly, the least contrast texturally between the seasonal subsamples occurs in B_5, which Dark and Allen (2005) suggest to mean a winter milder and less stormy than normal, followed by a summar cooler than usual. Supporting this interpretation, the isotope data for S_5 and W_5 also show little contrast. Bands B_2–B_4 show the strongest cyclical patterns in terms of C/N ratio, with $\delta^{13}C$ having less variation.

Our findings, based on a comparatively small number of samples, are preliminary, and caution should therefore be excercised in applying them to environmental reconstruction. Neverthless, because of known variations in the relative contribution of organic material from different sources to the Severn Estuary, our results from modern and seasonally banded estuarine silts suggest that isotope values and C/N ratios have the potential to (1) distinguish between sediments deposited at different times of year, and (2) hint at the quality of seasons and the extent of interannual variability. Future work will be directed at fully testing the seasonal changes in $\delta^{13}C$ and C/N ratios in sediments of salt-marsh origin as the result of high-frequency sampling over a sequence of years.

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