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Middle to Late Holocene Fluctuations of C$_3$ and C$_4$ Vegetation in a Northern New England Salt Marsh, Sprague Marsh, Phippsburg Maine

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**Key Words**: carbon isotopes, leaf wax biomarkers, sea-level, salt marsh, Gulf of Maine, Holocene
Abstract

A 3.1 meter sediment core was analyzed for stable carbon isotope composition of organic matter and higher plant leaf wax (HPLW) lipid biomarkers to determine Holocene shifts in C\textsubscript{3} (higher high marsh) and C\textsubscript{4} (low and/or high marsh) plant deposition at the Sprague River Salt Marsh, Phippsburg, Maine. The carbon isotope composition of the bulk sediment and the HPLW parallel each other throughout most of the core, suggesting that terrestrial plants are an important source of organic matter to the sediments, and diagenetic alteration of the bulk sediments is minimal. The current salt marsh began to form 2500 cal yr BP. Low and/or high C\textsubscript{4} marsh plants dominated deposition at 2000 cal yr BP, 700 cal yr BP, and for the last 200 cal yr BP. Expansion of higher high marsh C\textsubscript{3} plants occurred at 1300 and 600 cal yr BP. These major vegetation shifts result from a combination of changes in relative sea-level rise and sediment accumulation rates.

Average annual carbon sequestration rates for the last 2500 years approximate 40 g C yr\textsuperscript{-1} m\textsuperscript{2}, and are in strong agreement with other values published for the Gulf of Maine. Given that Maine salt marshes cover an area of ~79 km\textsuperscript{2}, they represent an important component of the terrestrial carbon sink. More detailed isotopic and age records from a network of sediment cores at Sprague Marsh are needed to truly evaluate the long term changes in salt marsh plant communities and the impact of more recent human activity, including global warming, on salt marsh vegetation.
Introduction

In New England salt marshes, the vegetation is zoned according to elevation of surface relative to mean high tide (summarized in Nixon, 1982; Teal, 1986; Bertness and Ellison, 1987; Bertness, 1991). The low marsh is closest to the tidal stream, and is located between mean high tide and the mean high water (MHW) level. It is inundated with salt water twice daily and is inhabited primarily by the tall form of *Spartina alterniflora* (C₄ plant; smooth cordgrass), a species that is well adapted to anoxic and saline conditions. The high marsh is located in the area above the mean high water level. It is flooded irregularly at high tides (e.g., spring tides) and is inhabited by a mosaic of *Spartina patens* (C₄ plant; salt meadow hay), *Distichlis spicata* (C₄ plant; spike grass), and the short form of *S. alterniflora* (C₄ plant). The higher high marsh is at or above mean higher high water (MHHW) and is flooded during big storm events. It is inhabited by increasing amounts of *Juncus* spp (C₃ plants), a plant with lower salinity tolerance than either *Spartina* spp. or *D. spicata* (Bertness, 1991), as well as *Scirpus* spp (C₃ plants). The brackish marsh borders the salt marsh and is generally dominated by *Scirpus* spp. and *Phragmites* spp. (C₃ plant; reed-grass) among other C₃ species.

The average carbon isotopic composition (δ¹³C) of salt marsh plants is -28.2 ± 2.3‰ (n=8) and -13.9 ± 1.2‰ (n=3) for C₃ and C₄ species, respectively. Therefore, salt marsh surface soil δ¹³C values should decrease from the low/high marsh to the higher high marsh, because of the increased C₃ plant biomass (e.g., *Juncus* spp. and *Scirpus* spp.) with increasing distance from the tidal channel (Figure 1; after Chmura and Aharon, 1995). Several have used this relationship as the basis for interpreting sedimentary organic matter (SOM) δ¹³C values in salt marsh cores. Isotopic changes reflect changes
in salt marsh plant communities which result from changes in relative sea level (RSL) through time (DeLaune, 1986; Chmura et al., 1987; Craft et al., 1988; Orson et al., 1998; Choi et al., 2001). These studies assume that salt marsh plants (i.e., autochthonous material) are the dominant source of organic matter to the sediments, and that algal biomass (i.e., allochthonous material) is not significant.

The $\delta^{13}C$ values of $C_3$ plants are similar to that of particulate organic matter (POM) in the Gulf of Maine, and the $\delta^{13}C$ values of $C_4$ plants are similar to certain macroalgae (McMahon et al., in press), both of which could be contributing substantially to organic matter deposition. Thus, independent proxies for identifying organic matter are needed to determine the relative importance of allochthonous and autochthonous deposition in the salt marsh. Recent lipid biomarker studies provide more specific information on cycling of plant organic matter within salt marsh sediments (Bull et al., 1999; Wang et al., 2003), but these studies lack a longer-term, geologic perspective from which valuable baseline information about salt marsh history can be obtained.

In the Gulf of Maine, the systematic use of carbon isotopes for reconstructing changes in $C_3$ and $C_4$ salt marsh plant communities has been secondary to the analysis of salt marsh peat rhizomes and salt marsh benthic foraminifera distributions for past sea level studies (Gehrels, 1994; 1999). We contend that given potential difficulties associated with rhizome and foraminifera identification and a lack of foraminifera in some settings, carbon isotopic investigations of Maine salt marsh cores could be useful for studies of RSL. Studies indicate that diagenetic modification of SOM in marshes is less than a few permil (Benner et al., 1987; Fogel et al., 1989; Chmura et al., 1987;
Ember et al., 1987), significantly less than the isotopic shifts accompanying shifts in salt marsh plant communities expected through time.

In this paper, we analyze the stable carbon isotope composition of bulk sediments and higher plant leaf wax (HPLW) biomarkers (i.e., the C\textsubscript{24}, C\textsubscript{26}, C\textsubscript{28}, C\textsubscript{30}, and C\textsubscript{32} n-alkanoic acids) extracted from a sediment core to reconstruct middle to late Holocene shifts in salt marsh plant communities in Phippsburg, Maine. The HPLW lipid biomarkers are ~7-10‰ depleted relative to the plant from which they are derived and are generally well preserved through geologic time (summarized in Brassell, 1993; Meyers, 1997), having been successfully recovered in samples as old as the Miocene (c.f., Freeman and Colarusso, 2001). The isotopic offset between the bulk sediments and the HPLW should be consistent in unaltered material dominated by terrestrial plant deposition due to the refractory nature of the HPLW. We use the isotopic offset between HPLW and bulk sediments to evaluate diagenetic alteration of the SOM at Sprague Marsh, and to evaluate changes in relative abundance of autochthonous and allochthonous organic material. Finally, we illustrate the importance of Maine salt marshes as a carbon sink by evaluating the long term carbon sequestration rates in the Sprague Marsh.

**Study Site**

The Sprague River Marsh is located within the Bates Morse Mountain Conservation Area in Phippsburg, Maine, at 43° 45’N/ 69° 50’W (latitude/longitude) (Figure 2). It is bordered on the east and west by two glacially carved north-south trending bedrock ridges, including Morse Mountain, and to the south by Seawall Beach, a barrier spit extending 2.2 km from the eastern rocky headland. The marsh is
approximately 2 km long and 800 m wide in the southern section and is drained by the Sprague River. There is a single-lane gravel causeway traversing the north end of the marsh that was put into place in the 1940’s. The coring site was approximately 15 m south of the causeway on the west side of the tidal channel (Figure 2).

Sprague Marsh is classified as a back-barrier marsh (after Kelley et al., 1988; Gehrels et al., 1996) in the south-central region of the Gulf of Maine (Jacobson et al., 1987). At the coring site, low marsh vegetation is found fringing the tidal channel and the ditches and is composed of the tall form of *S. alterniflora*. High marsh dominates the salt marsh surface, and is represented primarily by a mosaic of *S. patens* and *D. spicata* with lesser amounts of the short form of *S. alterniflora*. The higher high marsh is represented by the presence of *Juncus gerardii* in addition to *S. patens* and *D. spicata* with lesser amounts of *Solidago* spp. (C₃ plants) *Scirpus robustus*, and *Triglochin maritimum* (C₃ plant). The brackish marsh is dominated by *S. robustus*, *J. gerardii*, and *T. maritimum*. *Salicornia europaea* (C₃ plant) is present across the marsh, but represents a small portion of the total biomass.

**Methods**

**Sample Collection**

A vibracorer was used to extract a 3.1 m sediment core from the high marsh at the northern end of Sprague Marsh, in May, 2002 (Figure 2). The core was labeled SM02-VC1, and stored for several months at 4 °C prior to splitting and subsampling. Once opened, the sediments were logged and subsampled every 10 cm for bulk geochemical analyses and every 30 cm for lipid analyses.
Sample Preparation and Analysis

Bulk dry densities were determined by drying a 1 cc aliquot of sediment at 100 °C until a constant mass was achieved. Dried core sediments were homogenized with a mortar and pestle and sieved at 40 mesh. A subsample of the <40 mesh fraction was decalcified using 1N H$_3$PO$_4$ (after Weliky et al., 1983) to remove any trace amounts of carbonate that might have been present. The decalcified sediments were analyzed for bulk geochemical parameters (i.e., %OC, C/N, and $\delta^{13}$C).

To isolate the HPLW, between 1 and 10 g of the homogenized, <40 mesh fraction were Soxhlet extracted for ~24 hours in methylene chloride: methanol (2:1 by volume) (after Wakeham et al., 1988). The resultant total lipid extract was saponified in 0.5 N KOH in methanol and separated into neutral and acid fractions. The acid fraction was methylated with 3% BF$_3$ in methanol and the resultant fatty acid methyl esters (FAME) were analyzed and quantified by GC-FID (Agilent 6890N) in the Environmental Geochemistry Laboratory (EGL), Department of Geology, Bates College. Compound identification was undertaken using the GC/MS (Agilent 6890/MS 5973) in the Department of Chemistry, Bates College.

Carbon isotope measurements of bulk organic matter were made in the EGL at Bates College using a ThermoFinnigan Delta Plus Advantage stable isotope ratio mass spectrometer (IRMS) interfaced to a Costech elemental analyzer (EA) via the Conflo III combustion interface. Compound specific isotopic analyses were made using a Trace GC and GCIII combustion interfaced to the aforementioned IRMS.

The running conditions for all GC analyses were similar so results could be readily compared. Samples were injected isothermally at 250 °C and compounds were
separated on an HP-5MS column (30 m, 0.25 mm i.d., 0.25 μm film). The oven temperature program was as follows: \( T_1 = 55 ^\circ C \) (5 minute hold); \( T_2 = 180 ^\circ C \) (15 \(^\circ C/\)minute with 0 hold); and, \( T_3 = 310 ^\circ C \) (8 \(^\circ C/\)minute with 25 minute hold).

All stable carbon isotope values are reported in delta (δ) notation, in units of per mil (‰), where \( \delta^{13}C = \left[ \left( \frac{^{13}C/^{12}C}_{\text{sample}} \right) / \left( \frac{^{13}C/^{12}C}_{\text{standard}} \right) - 1 \right] \times 1000 \), and the standard is VPDB (Craig, 1957). The external reproducibility, as determined by the standard deviation of multiple analyses of the bulk sediment was ± 0.2‰, and of the individual FAME was ±1.0‰. The \( \delta^{13}C_{\text{HPLW}} \) is presented as the weighted mean according to the following equation:

\[
\frac{\delta^{13}C_{C_24}*[C_{C_24}]}{([C_{C_24}])} + \frac{\delta^{13}C_{C_26}*[C_{C_26}]}{([C_{C_26}])} + \frac{\delta^{13}C_{C_28}*[C_{C_28}]}{([C_{C_28}])} + \frac{\delta^{13}C_{C_30}*[C_{C_30}]}{([C_{C_30}])}
\]

where the brackets represent the concentration of the enclosed compound.

**Radiocarbon Chronology**

Three macrofossils were collected for radiocarbon analyses at three different depths within the micaceous sandy layer, including a small piece of wood at 274 cm, and a fragment of *Macoma balthica* at 253 cm and a fragment of *Mya arenaria* at 216 cm. An acid-base extraction was performed on the wood (after Tom Brown, pers. comm.), and the shells were mechanically abraded prior to a ~10% acid leach in 0.1N HCl. All samples were sent to the AMS \(^{14}C\) dating laboratory at the Livermore National Laboratory.

Radiocarbon dates were calibrated using CALIB 4.4 and are reported as the midpoint of the age range (2σ) with the largest relative probability in calibrated years before present (cal yrs BP) (after Stuiver et al., 1998). The marine reservoir
correction for the Gulf of Maine is about 400 years (Tanaka et al., 1990), but, as the mollusks dated in this study are intertidal, the full marine reservoir correction is not appropriate for these samples. On this basis, we have not applied any marine reservoir corrections before calculating the calibrated ages of the mollusk samples reported here. We note that the actual ages of the mollusk fragments may be up to 400 years younger than indicated by the reported radiocarbon dates, and further, that such shifts in the mollusk ages would have no significant impact on the preliminary core chronology nor on this study's conclusions.

**Results**

**Core Stratigraphy and Chronology**

Five stratigraphic units were differentiated from one another based on sediment color, texture, mineralogy and rhizome composition (Figure 3). These units include a lower organic rich layer of unknown origin between 298 and 276 cm, overlain by tidal flat muds and sands containing sporadic shell fragments, including the wood and *Macoma balthica* and *Mya arenaria* shell pieces used for radiocarbon dating. The tidal flat deposits grade into a rhizome rich salt marsh peat beginning at approximately 175 cm. Between 175 and 0 cm, fluctuations in rhizome morphology occur and are used to discern an overall regression in sea level, where low marsh at the bottom grades into mixed low/high marsh in the middle and high marsh at the top of the peat (Figure 3).

The radiocarbon dates increase with depth in the core, where the base of the intertidal sands is 3700 cal yrs BP (Table 1). An age-depth model was constructed by fitting a linear regression through the three radiocarbon-dated data points and the origin
The resultant average sedimentation rate is approximately 0.07 cm yr\(^{-1}\) and is in good agreement with modern sedimentation rates of high marshes in the Bay of Fundy, which range between 0.03 and 3 cm yr\(^{-1}\) (Connor et al., 2001). This sedimentation rate is used to assign ages to core depths above the lowest sample dated with the assumption that the marsh surface represents the present day, and that the rate of sedimentation has been constant through the last 3700 years. The assumption of constant sedimentation is unlikely given the difference in depositional environments and physical properties of the intertidal sands versus the salt marsh peat. Thus, the assigned ages should be considered preliminary until verified with additional AMS \(^{14}\)C dates from multiple units.

**Organic Geochemistry**

The lowest %OC values are measured in the tidal flat deposits and the highest values are measured in the salt marsh peat and organic rich sediments (Table 2; Figure 4). Down-core variations in the %OC likely results from a combination of factors, including changes in organic matter sources, changes in inorganic sediment fluxes, and/or changes in rates of organic matter decomposition at the time of burial.

The atomic C/N values of the core sediments range between 14 and 22, and are typical of terrestrially derived organic matter (Meyers, 1997) (Table 2; Figure 4). These elevated C/N values are not particularly surprising in the peat sections of the core where terrestrial materials (i.e., rhizomes) are abundant. High C/N values found in the middle of the intertidal sand and silt deposits, however, indicate that terrestrial plant deposition was significant in the nearshore tidal flats as well.

The \(\delta^{13}\)C values of the bulk organic matter range between -13 and -25 \(^\circ\)o, and the weighted \(\delta^{13}\)C\(_{HPLW}\) values range between -20 and -31\(^\circ\)o (Table 2; Figure 4). The HPLW
isotope data are consistently depleted in $^{13}$C by ~7‰ relative to the bulk isotope data and are correlated strongly to each other, where $\delta^{13}$C$_{HPLW} = 0.9 \delta^{13}$C$_{bulk} - 7.5$ (R$^2 = 0.90$) at all depths except two (270 and 177 cm). This isotopic consistency between the bulk sediment and the HPLW implies that the bulk isotope data have not been altered significantly by diagenesis, and that the data reflect a terrestrial plant source throughout most of the core.

At 270 and 177 cm, the average isotopic offset between the HPLW and the bulk sediments is 3.1 and 1.7‰, respectively. This reduced offset implies that the bulk sedimentary organic matter (1) has been significantly modified by diagenesis, (2) is derived from aquatic sources that are enriched in $^{13}$C, such as the macroalgae *Ascophyllum nodosum* [$\delta^{13}$C = -17.3 ± 0.4‰ (n=3); McMahon et al., in press], or (3) is dominated by HPLW derived from C$_3$ plants. High C/N values and relatively high HPLW concentrations (Table 2) favor the third scenario listed above; however, a detailed analysis of the HPLW concentration data from the sediments, modern plants and other possible organic sources to the site is needed to corroborate this interpretation.

**Discussion**

**Middle to Late Holocene Environments of Deposition**

The basal unit of the core is an organic rich mud layer without rhizomes which appears to correlate with a similar unit of unknown origin noted in other cores from the same general area (Gehrels et al., 1996). The enriched carbon isotope composition of the bulk sediments and the HPLW provides new insights into the origin of this unit, and indicates that it contains a significant amount of C$_4$ plant material, and represents a period
of low and/or high marsh plant colonization. The absence of rhizomes suggests that the organic matter may have been reworked into the sedimentary sequence above as sea level rose.

Deposition of the intertidal sands and mudflats overlying the lower organic unit began approximately 3700 cal yr BP. This unit is relatively thick and widespread at Sprague Marsh, and may indicate a period of rapid sea level rise (RSLR) (Gerhels et al., 1996) and reworking of the earlier salt marsh unit.

An organic rich, salt marsh peat overlies the intertidal sands and extends from 175 cm to the surface. Assuming a linear sedimentation rate of 0.07 cm yr$^{-1}$ over the dated region of the core, the peat began to form 2500 yrs BP, and is in agreement with other studies at Sprague Marsh (Gehrels et al., 1996). This shift from intertidal deposits to salt marsh deposits coincides with a reduction in rates of RSLR that occurs between 2500 and 1000 cal yr BP (Gehrels, 1994).

The isotopic composition of the upper peat reflects the following conformable salt marsh communities from oldest to youngest: low and/or high marsh (C$_4$ plants) grading into higher high marsh (C$_3$ plants) grading into low and/or high marsh (C$_4$ plants) grading into higher high marsh (C$_3$ plants) grading into the present high marsh (C$_4$ plants). These multiple shifts in plant communities were not identified in the rhizome stratigraphy presented herein, nor in the stratigraphy presented by others (Gehrels et al., 1996). These results illustrate the added value of carbon isotope data for discerning changes in salt marsh plant communities through time.

At Sprague Marsh, higher high marsh plant maxima occur at 1300 and 600 cal yr BP and high and/or low marsh plant maxima occur at 2000, 700, and 200 cal yr BP. The
balance between RSLR and sediment accumulation is the primary environmental factor determining long term changes in marsh structure (DeLaune et al., 1983; DeLaune, 1986; Warren and Niering, 1993) and depends on a variety of factors, including sediment supply, flooding regime, compaction, and plant community structure (Niering and Warren, 1980; Orson et al., 1998). C4 marsh forms when RSLR exceeds sediment accumulation rates and C3 marsh forms when sediment accumulation rates exceed RSLR.

Additional environmental factors to consider when interpreting C3 and C4 vegetation shifts in salt marshes include ice rafting, storms and human activity. Over the last 300 years, for example, humans have altered the hydrology of Northern New England salt marshes dramatically (via salt marsh hay and cranberry production, causeway installation and ditching for mosquito and greenhead fly control), and little is known about the consequences of these actions on salt marsh structure.

Carbon Sequestration

The long-term average carbon sequestration rate of the Sprague River salt marsh is ~40 g C yr\(^{-1}\) m\(^{-2}\) and was calculated as the product of the average dry bulk density (0.28 g cm\(^{-3}\)), the fraction of sediment comprised of OC (0.20), and the average sedimentation rate (0.07 cm yr\(^{-1}\)) in the upper peat unit (i.e., upper 175 cm). This value is within an order of magnitude of estimates of recent carbon sequestration rates determined for high salt marsh sediments in the Bay of Fundy, eastern North America (Chmura et al., 2003) and southern California (Brevik and Homburg, 2004). Maine salt marshes cover an area of ~79 km\(^2\), more than any other state north of New Jersey (Jacobson et al., 1987), therefore their importance as a carbon sink should not be overlooked.
It has been argued that a continued rise in sea level brought about by global warming would actually increase the terrestrial carbon sink by expanding salt marshes onto gently sloping upland terrain, thus modulating future global warming and sea level rise (Choi et al., 2001). Sprague Marsh, and many others in the south-central region of the Gulf of Maine, occupy glacially carved valleys and are surrounded by topographic highs, and will likely behave differently than those studied by Choi et al. (2001). A rapid sea level rise in the Gulf of Maine might result in drowning of the salt marshes and loss of an effective carbon sink.

Conclusions

In Phippsburg Maine, terrestrial plant organic matter has been a significant component of coastal sediments for the last 3700 years. High C/N values and depleted δ¹³C of the HPLW suggest that C₃ plant detritus was an important part of nutrients to the food web in the middle Holocene tidal flats at Sprague Marsh as well as the overlying salt marsh peat.

Stable carbon isotopes of salt marsh peat cores are useful indicators of low and/or high marsh (C₄ dominant Spartina spp.) and higher high marsh (C₃ dominant Juncus spp.) plant communities when rhizome identification is equivocal and foraminifera identification is not possible. The isotope data suggest that vegetation has responded to small changes in relative local sea level (i.e., two transgressional-regressional sequences followed by a transgressional sequence) over the last 2500 years. These shifts represent changes in the balance between RSLR and sediment accumulation.
High resolution sea level records suggest a strong correlation between Greenland bore hole temperatures and relative sea level along the Maine Coast over the last 2000 years (Gehrels et al, 2002). Different responses have been documented in salt marshes (Choi et al., 2001; Donnelly and Bertness, 2001). High resolution isotopic studies of salt marsh cores covering the last 2000 years would assist in our understanding of and ability to predict salt marsh responses to global warming and sea level change. A larger network of sediment cores, each with better age control and more isotopic data, would be necessary to determine the response of the Sprague Marsh to these various environmental conditions.

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References Cited


List of Figures

Figure 1. Generalized cross section of a New England salt marsh showing major vegetation types, where Sat = tall *Spartina alterniflora*; Sp = *Spartina patens*; Ds = *Distichlis spicata*; Sas = short *Spartina alterniflora*; Se = *Salicornia europaea*; If = *Iva Fructescens*; Jg = *Juncus gerardii*; Pv = *Panicum virgatum*; Pa = *Phragmites australis* [figure modified from Neiring and Warren (1980)].

Figure 2. The Sprague River Marsh, in the Bates-Morse Mountain Conservation Area, Phippsburg, Maine. The stippled areas represent water, where the Sprague River runs N/S in the middle of the marsh, and joins the Atlantic Ocean to the SW of Seawall Beach. The dark lines in the marsh represent a combination of the natural meanders of the Sprague River channel prior to dredging, and human made ditches. Arcview (ESRI, Inc. Redlands, CA) was used to generate the map and electronic data for the coastline, river and road were obtained from USGS 1:24,000 scale quadrangle maps obtained from the Maine Office of GIS website ([http://apollo.ogis.state.me.us/](http://apollo.ogis.state.me.us/)).

Figure 3. Sprague marsh core stratigraphy, radiocarbon ages and age model. The three samples that were radiocarbon dated are as follows: A = *Mya arenaria*; B = *Macoma balthica*; and, C = wood fragment.

Figure 4. Organic geochemical data from Sprague Marsh core, including percent organic carbon (%OC), C/N atomic values (C/Nat), bulk sediment organic carbon isotope data ($\delta^{13}C$ Bulk) and higher plant leaf wax isotope data ($\delta^{13}C_{HPLW}$, presented as the weighted mean for C24, C26, C28, C30, and C32). Ages assigned based on age model (Figure 3) and sample depth.
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Table 1. Radiocarbon and calibrated ages for three samples collected from core SM02-VC1.

Table 2. Organic geochemical data for the Sprague Marsh sediment core, SM02-VC1.
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Figure 1
Figure 3

y = 0.07x - 1.34
(R^2 = 0.98)

Stratigraphic Key

- High marsh peat
- Mixed high and low marsh peat
- Low marsh peat
- Tidal flat mud
- Organic rich mud