

ACCURATE LACUSTRINE AND WETLAND SEDIMENT ACCUMULATION RATES DETERMINED FROM ^{14}C ACTIVITY OF BULK SEDIMENT FRACTIONS

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ABSTRACT. In the absence of identifiable macrofossils in lacustrine sediments, radiocarbon dating must rely on pollen or bulk sediment fractions. Bulk sediment fractions are not generally preferred because they contain an unknown mixture of organic material of variable age, they may contain dead carbon such as lignite that is difficult to eliminate, and material of aquatic origin may be subject to reservoir effects. If the various processes that contribute carbon to the system are relatively constant over time, however, changes in ^{14}C activity with depth may be used to accurately estimate sediment accumulation rates even if the absolute ages are erroneous. In this study, fine-grained fractions (250–710 μm organic material, humic acids extracted from <250- μm fraction, and untreated <250- μm fraction combusted at low temperature) were analyzed and compared with terrestrial plant stems (twigs), charcoal, and wood fragments in sediments from an oxbow lake in Mississippi, USA. The ^{14}C activities of the bulk fractions were highly linear with depth and produced consistent calculated sediment accumulation rates similar to, and perhaps more reliable than, rates determined using twigs or charcoal.

INTRODUCTION

Radiocarbon is commonly used to date individual sediment layers or to estimate ancient sedimentation rates in lacustrine sediments. Ideally, the target material for dating is clearly identifiable terrestrial macrofossils. If a terrestrial organism is washed into a lake shortly after death, the age of the preserved fossil will be approximately concurrent with the date of deposition. Aquatic organisms that die and settle on the lake bottom may more precisely represent the age of sediment deposition, but these organisms are subject to reservoir effects that can yield falsely old ^{14}C ages (Björck and Wohlfarth 2001).

In low-organic lacustrine sediments, macrofossils can be difficult to find. In these sediments, various bulk sediment fractions or pollen may be used for dating. Pollen has the advantage of being ubiquitous in lacustrine environments, but dating often requires large sediment samples. Isolation or concentration of pollen can also be labor intensive and requires the use of hazardous chemicals (Brown et al. 1992; Bennett and Willis 2001; Vandergoes and Prior 2003). Various bulk sediment fractions, obtained by sieving or chemical separation, are not typically preferred for dating because of several potential problems. First, the organic fraction represents a mix of material of varying age that could include remains of burrowing organisms or roots from aquatic plants (younger than age of sediment deposition), terrestrial material that spent many years on land before being washed into the lake (older than age of sediment deposition), or ancient material such as lignite or graphite (older than age of sediment deposition). Second, aquatic organisms contributing organic material to the sediment are subject to reservoir effects that will typically add apparent age to ^{14}C dates. Reservoir effects may be caused by incorporation of carbon derived from calcite dissolution, decomposition of ancient organic matter, groundwater discharge, or volcanic emissions (Björck and Wohlfarth 2001).

Numerous investigations have been performed on different lacustrine bulk sediment fractions to determine if any yield ^{14}C ages representative of the time of deposition. The majority have compared humic acids (alkali soluble) with untreated, acid-treated, or acid-alkali-treated (alkali insoluble)

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ble) bulk sediment. Most have reported younger ^{14}C ages for humic acids relative to the other bulk sediment fractions, but it is not always certain which fraction most closely represents the time of deposition. As examples, studies reported by Olsson (1991), Björck et al. (1994, 1998), Barnekow et al. (1998), and McGeehin et al. (2001) concluded that humic acids were most representative of depositional age, while Walker and Harkness (1990), Åkerlund et al. (1995), and Hedenström and Risberg (1999) gave preference to the alkali-insoluble fraction.

Broader studies including additional organic fractions have produced more variable results. Abbott and Stafford (1996) reported ^{14}C ages of sediment lipids to be younger than humic acids, while lipid ages reported by Fowler et al. (1986) were often older than humic acids or acid-treated bulk sediment. Studies in soils on a wide range of organic fractions have produced similarly equivocal results. Becker-Heidmann et al. (1988) and Pessenda et al. (2001) found untreated bulk sediment to yield the youngest ages relative to charcoal and alkali-insoluble fractions at most depths. Scharpenseel and Becker-Heidmann (1992), reviewing 25 yr of soil dating efforts, concluded that a single sediment fraction could not be identified to yield reliable ages of soil formation independent of other supporting evidence.

Uncertainty associated with the identity and origin of carbon in bulk sediments limits the ability to determine an absolute age of a specific sediment layer, but it does not preclude the potential to determine accurate accumulation rates. If the various processes supplying organic material to a lake remain approximately constant over a period of time, bias in the ^{14}C activity introduced by reservoir effects or by mixing of inwashed material of different ages will be approximately constant as well. The change in ^{14}C activity with depth could then be used to determine the time span represented by a given thickness of sediment, yielding an accurate rate of sediment accumulation even if the absolute date of each measured layer is uncertain.

In this study, the ^{14}C activity of several sediment fractions were compared to determine if some or all would yield reliable sediment accumulation rates for sediments in an oxbow lake-wetland in Mississippi, USA. Fractions studied included: 1) terrestrial plant stems (twigs) representing 1 to 2 yr of growth; 2) wood fragments; 3) charcoal; 4) bulk sediment between 250 and 710 μm (mostly organic); 5) humic acids leached from the <250- μm fraction (silt and clay); and 6) untreated silt and clay (<250 μm) combusted at low temperature.

STUDY SITE

Lacustrine and wetland sediments were collected from Sky Lake, an oxbow lake-wetland located approximately 10 km north of Belzoni, Mississippi, USA (Figure 1). Sky Lake is an ancestral meander loop of the Mississippi-Ohio River system, estimated to have been abandoned between 7500 and 10,000 BP (Saucier 1994). The lake sits atop the Mississippi River alluvium, which consists of clays, silts, sands, and gravels with a local thickness between 100 and 150 ft (Arthur and Strom 1997). The Mississippi River alluvium and underlying unconsolidated sediments are relatively free of calcareous deposits.

The watershed draining into the lake is approximately 1900 ha of agricultural land. The region surrounding the lake was forested until clearing for agricultural use began in the late 19th century (Davidson et al. 2004). The perimeter of the lake continues to support a forested wetland up to 1 km in width, dominated by bald cypress (*Taxodium distichum*), waterlocust (*Gleditsia aquatica*), and water tupelo (*Nyssa aquatica*). The central open-water portion of the lake has a flat muddy bottom virtually free of vegetation. Water depths vary seasonally, with winter highs up to ~4.5 m and summer lows often falling below 0.5 m. The wetland is fully inundated when water depths in the lake

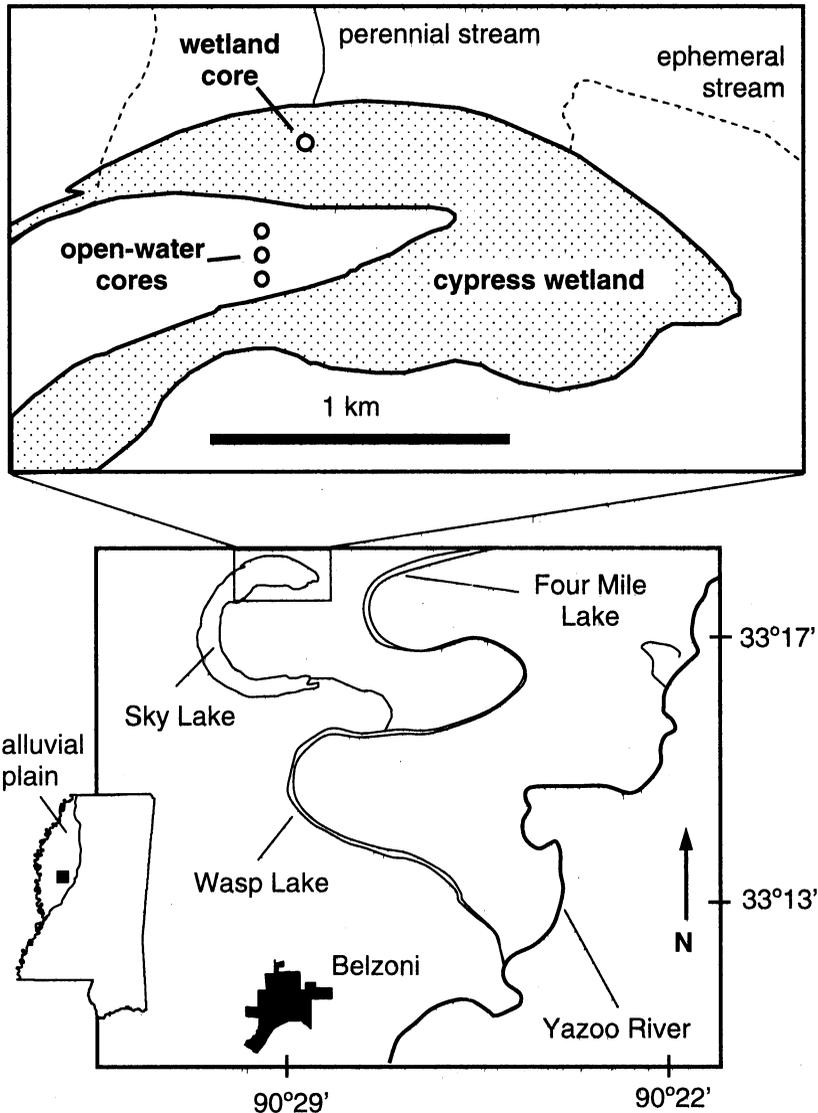


Figure 1 Study site: the alluvial plain is the ancestral floodplain of the Mississippi-Ohio River system.

reach approximately 1.5 m. Sky Lake is hydrologically perched above a regional water table that lies well below the lake bottom (Arthur 2001). Prior to heavy pumping for irrigation, the regional water table was higher (Fisk 1944; Brown 1947) and may have had periodic direct connection with the lake, though still serving as a source for groundwater rather than a sink.

Sediment transport to the lake occurred historically via runoff from several perennial and ephemeral streams, and via backflow when floodwater from the Yazoo River entered through Wasp Lake. Flow control structures installed in the 1980s now reduce backflow from the Yazoo River.

METHODS

A single sediment core was collected from within the forested wetland at the northern end of Sky Lake (Figure 1) for comparison of ^{14}C activities obtained from different sediment fractions. Three cores were collected from the unvegetated, open-water region to determine if estimates of sediment accumulation rates using ^{14}C activity from one of the bulk sediment fractions (low-temperature combustion of the $<250\text{-}\mu\text{m}$ fraction) would yield reproducible results.

The wetland core was collected by hammering a 10-cm-diameter PVC pipe to a depth of 1.5 m. Compression caused by coring was determined by measuring the depth to the top of the core inside the core barrel every 10 cm of penetration depth. The core was extruded in the laboratory and sectioned into wafers 2.3 cm in thickness. The outer edge of each wafer was cut away to eliminate mixed material smeared along the inside of the core barrel during collection. The open-water cores were collected using a vibra-coring system and 7.5-cm-diameter aluminum irrigation pipe to a depth of approximately 2.8 m. Compression caused by coring was determined by measuring the final depth of penetration and the length of sediment collected in the core barrel. These cores could not be extruded with available equipment and were cut on either side to remove the core barrel. Intervals for ^{14}C analysis were sectioned into wafers 1 cm in thickness.

Sediment accumulation rates reported by Davidson et al. (2004) were used to ensure that samples selected for this study predate settlement and clearing of the surrounding land. Woody material was pretreated using an AAA-bleach procedure for removing potential contaminants and reduction to holocellulose (adapted from Hoper et al. 1998). Samples were placed sequentially in 1M HCl for 2 hr at 60 °C; 0.5M NaOH at 60 °C for 1 hr (repeated until solution was clear); 1M HCl for 1 hr at 60 °C; and a bleaching solution of 0.3M NaClO₂ and 0.07 M HCl at 60 °C until the material turned white (typically 4–8 hr). Samples were rinsed with distilled water between each step.

Samples consisting primarily of fine organic material (250–710 μm) were completely degraded when applying the full AAA pretreatment, so a less aggressive pretreatment was performed on these samples. The first acid wash was limited to 1 hr and only 1 alkali wash was performed. Duplicate samples were combusted without pretreatment to determine the importance of pretreatment for these samples.

With the exception of the untreated $<250\text{-}\mu\text{m}$ fraction, all samples were combusted in quartz tubing with excess CuO over an open propane flame. Evolved gases were passed through hot Cu and Ag wire to convert nitrogen oxide gases to N₂ and to remove residual halides.

The $<250\text{-}\mu\text{m}$ fraction (silt and clay) was split into 2 subsamples. One split was pretreated following the first 2 steps of the AAA pretreatment above and the leachate from the alkali step saved. The leachate was acidified to pH 2 to precipitate humic acid and centrifuged. The solid precipitate was rinsed with deionized water, dried, and combusted as described above. The second split was combusted untreated at a temperature of 400 °C with O₂, following the procedure outlined in McGeehin et al. (2001), to avoid releasing CO₂ bound in the clay mineral structure (Delqué Kolić 1995). Evolved gasses and O₂ were passed first through quartz beads heated to 1000 °C to convert residual CO to CO₂, and through hot Cu and Ag as above.

CO₂ for all combustions was purified cryogenically and split into fractions for $\delta^{13}\text{C}$ and ^{14}C analyses. Graphite targets for ^{14}C analysis were prepared by conversion of CO₂ to graphite in the presence of powdered Zn and Fe (Slota et al. 1987), and analyzed by accelerator mass spectrometry (AMS) at the NSF-Arizona AMS facility.

Sediment accumulation rates were calculated for each sediment fraction by plotting the natural logarithm of the ^{14}C activity versus sample depth, calculating a best-fit line through the data, and multiplying the slope of the line by the decay constant of ^{14}C .

RESULTS AND DISCUSSION

Sediment Characterization

Twigs, wood, and charcoal larger than 710 μm were found with some regularity in wetland sediment cores, but were absent in sediment collected from the open-water environment with the exception of a few isolated wood fragments. In both wetland and open-water environments, inorganic sediments were limited to clay- and silt-size particles <250 μm , with approximately 40% by volume less than 45 μm . The organic content of sampled sediment was generally <10%. (Higher organic contents reported in Davidson et al. [2004] were in modern sediments deposited following land clearing.)

Sediment Fractions from the Same Wetland Core

Ideally, calibrated age probability distributions should be created from the ^{14}C activities at each depth, and sedimentation rates determined by fitting curves that maximize intersection with the ages of highest probability (Reimer and Reimer 2006; Bronk Ramsey, forthcoming). This approach is appropriate for discrete macrofossils, but will yield less meaningful results for bulk sediment fractions. The ^{14}C activity measured on bulk samples represents an average activity contributed from a variety of different sources and representing a range of actual ages. A distribution of possible calibrated ages derived from an average ^{14}C activity will not be the same as the summed distribution of possible ages for each individual organic particle in the sample. For this reason, sediment accumulation rates are calculated here using the raw ^{14}C activities and assuming changes in activity can be directly converted into an equivalent span of time. We assume that in an idealized system, where all processes and inputs are constant over time, the natural logarithm of activities with respect to depth will fall on a straight line. Major long-term wiggles at plateaus in the calibration curve could make this assumption invalid.

A comparison of activity-depth plots for each sediment fraction from the wetland core is shown in Figure 2. The activities of discrete subfossil samples (twigs, charcoal, and wood) show much more scatter with depth than the bulk sediment fractions (fine organic debris 710–250 μm ; humic acids from the <250- μm fraction; and the untreated <250- μm fraction combusted at low temperature). Scatter is to be expected in the activity of wood fragments, because decaying tree trunks and limbs may not be transported and deposited in sediments until decades after death. Less scatter is expected for the twig and charcoal samples, which should be transported and deposited much closer to the time of death or burning. Scatter for the twig data, however, is as great as the wood data ($r^2 = 0.76$ and 0.75 , respectively; Table 1). The charcoal data is more linear ($r^2 = 0.89$), but significantly less than the bulk sediment fractions ($r^2 = 0.96$ to 0.99).

Poor linearity is not due to historical variation in atmospheric production of ^{14}C , because the twig and charcoal activities appear to vary independently, and conversion of these activities to calibrated dates does not improve linearity with respect to depth. These data indicate that the sediments were either partially reworked after deposition or that there was considerable variability in the time the sampled material resided in the terrestrial environment before transport to the lacustrine-wetland sediments. The high degree of linearity among the bulk sediments suggests that significant reworking has not taken place. Incorporation of material of a variety of ages, however, is supported by the bias in the bulk sediment fractions toward older apparent ages (lower activities).

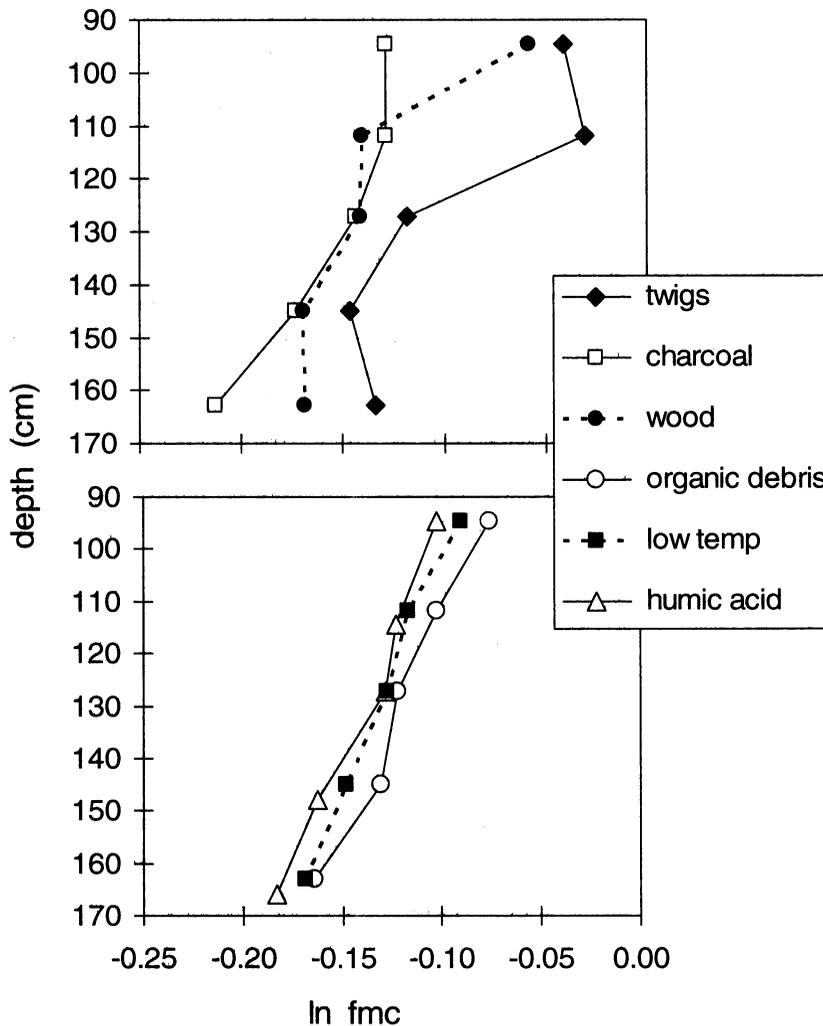


Figure 2 ¹⁴C activity of samples from the wetland core (Figure 1). Data are plotted in 2 separate graphs so all data points are visible: discrete samples (twigs, charcoal, and wood fragments) are plotted in the upper graph and bulk sediment fractions (250–750- μ m fraction – “organic debris”; humic acid extract from <250- μ m fraction – “humic acid”; and untreated <250- μ m fraction combusted at low temperature – “low temp”) are plotted in the lower graph. Pretreated and untreated organic debris were not significantly different, and only the pretreated results are plotted (ln fmc = natural logarithm of fraction modern carbon).

At most depths, twigs yielded the youngest apparent ages and charcoal the oldest. The bulk fractions were generally intermediate between the twigs and charcoal, with the apparent age decreasing from organic debris (710–250 μ m), to the untreated silt and clay fraction (low-temperature combustion), and finally to the humic acids fraction. The greater apparent age of the humic acids contrasts with the results reported by most lacustrine studies cited in the Introduction. The organic debris was processed both with and without AAA pretreatment, with no significant difference in ¹⁴C activity or change in slope (Table 1).

Table 1 Linearity (r^2), slope (cm depth/ \ln fraction modern carbon), and calculated sediment accumulation rate based on plots of \ln of ^{14}C activity versus depth for each sediment fraction.

Location and sediment fraction	r^2	Slope (cm/ \ln fmc)
Wetland core (Figure 2)		
twigs	0.76	428
charcoal	0.89	707
wood	0.75	506
710–250 μm (organic debris – pretreated)	0.97	810
710–250 μm (organic debris – untreated)	0.96	855
<250 μm , humic acid extract	0.98	849
<250 μm , low-temperature combustion	0.99	894
Open-water cores (Figure 3)		
all <250 μm , low-temperature combustion	r^2	Slope (cm/ \ln fmc)
Core	Data points^a	
1	upper 4	0.99 170
2	upper 4	0.999 133
3	upper 3	0.93 109
1	lower 2	553
2	lower 2	1376
3	lower 3	0.82 791

^a“upper” and “lower” data share the transition point.

Bias in the activity of individual bulk sediment samples and scatter in the discrete sediment samples make the use of any single sample to determine an absolute age in these sediments questionable. The high degree of linearity among the bulk fractions and similarity in slope, however, suggests that the processes introducing bias have been relatively constant over time in this system, making it possible to calculate meaningful sediment accumulation rates. Calculated rates of sediment accumulation based on the slope of best-fit lines for the data in Figure 2 range from 1.0 to 1.1 mm/yr for the bulk sediment fractions, and from 0.5 to 0.9 mm/yr for the twigs, charcoal, and wood (Table 1).

Open-Water Cores: Low-Temperature Combustion of Silt and Clay Fraction

All 3 of the bulk fractions analyzed in the wetland core appear to yield reliable data for estimating sediment accumulation rates. One fraction was chosen to determine if reproducible results could be obtained from multiple cores in the same depositional environment. In most low-organic sediments, the nearly pure organic fraction between 250 and 710 μm found in the wetland core will not be present. Of the remaining 2 bulk fractions, the low-temperature combustion of the silt and clay fraction was chosen over humic acids because of greater simplicity in sample preparation.

^{14}C measurements from 5 depths were obtained for each of 3 different open-water cores (Figure 3). All 3 cores show a high degree of linearity for \ln of ^{14}C activity versus depth for points above 180 cm (Table 1), and a consistent change in slope for the underlying points. The slopes for the upper data yield sediment accumulation rates ranging from 0.13 to 0.21 mm/yr. The lower points with greater slope yield sediment accumulation rates ranging from 0.67 to 1.66 mm/yr.

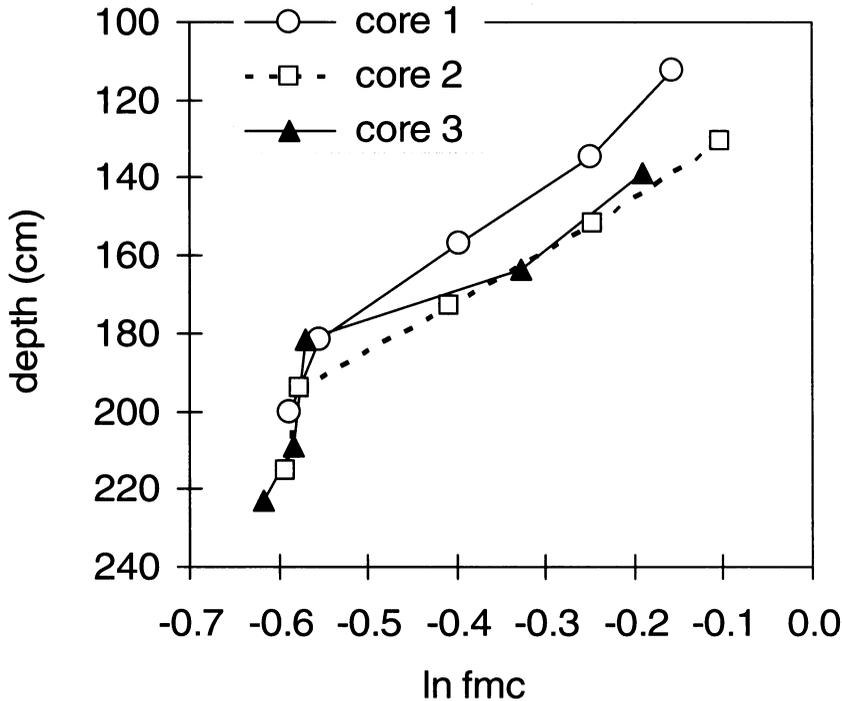


Figure 3 ^{14}C activity of samples from the 3 open-water cores (Figure 1). All data is untreated <250- μm fraction combusted at low temperature (ln fmc = natural logarithm of fraction modern carbon).

The change in rate is consistent with what is known of the evolution of the lake. The deepest samples were collected approximately 10 cm above coarse sands that filled the bottom of all 3 cores. The sands probably represent channel deposits when Sky Lake was part of the river. When an oxbow is first abandoned, accumulation of silts and clays will be relatively high due to intermittent flooding from the adjacent river. As the river migrates farther from the oxbow, the primary source of sediment shifts to runoff from the surrounding land and accumulation rates are lower as reflected by the upper data points.

CONCLUSIONS

Calculated sediment accumulation rates based on the ^{14}C activity of 3 bulk sediment fractions (undifferentiated organic debris between 250 and 710 μm , humic acid extracted from the <250- μm fraction, and the untreated <250- μm fraction combusted at low temperature) from the same wetland core ranged from 1.0 to 1.1 mm/yr. Rates based on the activity of twigs, charcoal, and wood fragments from the same depths ranged from 0.5 to 0.9 mm/yr. Rates determined using the twigs or charcoal would normally be considered the most reliable because terrestrial samples are less prone to suffer from reservoir effects, are likely to have been washed into the lacustrine or wetland environment shortly after death or charring, and contaminants such as calcite or lignite can be eliminated. The results of this study suggest that under certain conditions, bulk sediment fractions may yield the more reliable rates.

Scatter in the ^{14}C activity with respect to depth for twigs and charcoal suggests that the discrete samples selected for analyses were deposited in the lake-wetland after variable residence times in the

terrestrial environment. A large degree of scatter in a small data set means that multiple lines could represent the data, with the “best-fit” line merely representing the closest fit to the limited data set. Collection of additional data will likely result in a shift in the position of the best-fit line. Bulk fractions also include material of varying age, but each sample represents an average activity, which minimizes bias introduced by a single particle. In this study, the bulk sediment fractions all produced highly linear plots with very similar slopes, and thus similar calculated sediment accumulation rates.

Sediment accumulation rates based on low-temperature combustions of fine-grained sediments from 3 organic-poor cores in the open-water region of Sky Lake ranged from 0.13 to 0.21 mm/yr for the upper data and 0.66 to 1.67 mm/yr for the lower data. The change in rate is consistent with expectations for an oxbow lake that became increasingly isolated from the parent river.

Use of fine-grained bulk fractions to determine sediment accumulation rates has the potential to work in any system where contributions from different sources have been relatively constant over an extended interval of time. Elimination of larger particles minimizes the chances of a single aberrant particle significantly altering the measured activity. The activity of an individual bulk sediment sample may always be of questionable use for determining an absolute age of a particular horizon, but changes in activity with respect to depth may nonetheless produce accurate rates of sediment accumulation.

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