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DATING OF PREHISTORIC BURIAL MOUNDS BY ^{14}C ANALYSIS OF SOIL ORGANIC MATTER FRACTIONS

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ABSTRACT. Dating of prehistoric anthropogenic earthworks requires either excavation for archaeological artifacts or macroscopic organic matter suitable for ^{14}C analysis. Yet, the former, in many cases, is undesirable and the latter is difficult to obtain. Here we present a soil science procedure, which has the potential to overcome these problems. It includes careful sampling of buried former soil surfaces, acid-alkali-acid fractionation of soil organic matter (SOM), and subsequent ^{14}C AMS dating. To test the procedure, soil from one of the largest known burial mounds in Scandinavia, Hohøj, and 9 other Danish burial mounds were sampled. The ^{14}C dates from extracted SOM fractions were compared to reference ages obtained by other methods. We show that humic acid fractions in 7 of the 10 mounds had the same age as the reference, or were, at maximum, 280 yr older than the reference ages. The best age estimates were derived from an organic-rich layer from the upper cm of buried soil or sod. Differences among SOM fraction ages probably indicate the reliability of the dating. Hohøj dated to approximately 1400 BC and, thus, was up to 500 yr older than other dated Scandinavian mounds of comparable size. The remaining investigated burial mounds were dated to between 1700 and 1250 BC. We conclude that combined sampling of buried soil surfaces, SOM fractionation, and ^{14}C analysis allows for dating of archaeological earthworks when minimal disturbance is required, or if no macroscopic organic remains are found.

INTRODUCTION

As most preserved historic and prehistoric anthropogenic earthworks in Europe are protected by law, a dating procedure involving minimal disturbance is required. Dating of the soil organic matter (SOM) seems attractive, since many anthropogenic constructions contain buried soil surfaces or sods with moderate or high carbon (C) content. However, dating of bulk samples, particle size fractions, and organic matter fractions from soil samples has been carried out with varying degrees of success in the last 20–25 yr (Scharpenseel and Becker-Heidmann 1992; Wang et al. 1996). Also, chemical fractionation had not been satisfactory in separating labile and refractory soil organic carbon (SOC) pools (Wang and Hseih 2002). However, one procedure has apparently been able to date the burial time of former soil surfaces with some success (Matthews 1980; van Mourik et al. 1999; Dalsgaard and Odgaard 2001). It involves chemical fractionation (acid-alkali-acid extraction) of the SOM prior to ^{14}C analysis and has, so far, yielded the best results in acid, aerobic, sandy soils.

The potential of yielding a reliable dating of soil depends on the turnover time of the SOC, which again is a function of the soil type, depth, acidity, land use, and redox potential. Present knowledge unanimously shows that ^{14}C dating of bulk SOM from different depths below the surface does not give an exact numerical age because SOM consists of a continuum of organic materials in all stages of decomposition (Scharpenseel and Becker-Heidmann 1992). ^{14}C dating of organic-rich surface layers (humus layers) containing high contents of C, which originates from plant material with a high decomposition rate, will give a close estimate of the burial time, whereas ^{14}C dating of A-horizon or subsoil overestimates the time of burial, as it merely relies on the SOC's protection against decomposition. In an archaeological context, this means that the measured ^{14}C date of the time of burial will appear too old. In archaeological excavations, buried humus layers or plant fragments are

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rare compared to well-preserved A-horizons or organic-containing surface layers and a method suitable for dating of archaeological earthworks should preferably be able to rely on such horizons alone.

The approximately 100,000 prehistoric burial mounds recorded in south Scandinavia typically consist of numerous sods turned upside-down, and were often constructed on top of an intact soil surface. In the other parts of northern Europe, the number of burial mounds with sods or buried surfaces is probably high, too. Dating the time of construction of prehistoric burial mounds has, in most cases hitherto, been based on archaeological findings and macroscopic organic remains and absolute dating of the construction time of mounds is rare. Furthermore, from previous reviews of soil science in archaeology, it has been concluded that ^{14}C dating of SOC—if it has been mentioned at all—is less useful (Scrudder et al. 1996; Hedges 1994). This conclusion was most probably associated with the problems of selecting an extraction procedure and the estimation of an SOC mean residence time, which makes interpretation difficult.

We hypothesize that the time of burial of a mound can be approximated by ^{14}C analysis of the different components of SOM in a buried soil surface after suitable chemical fractionation, since the SOC was probably formed via photosynthesis from atmospheric carbon dioxide at, or shortly before, the time of burial. Hence, the aims of the present study were: i) to test the usefulness of a chemical fractionation of SOM in an archaeological context, and ii) to estimate the time of construction of 10 selected Danish burial mounds with minimal disturbance.

MATERIALS AND METHODS

Field sites and sampling

Two separate investigations were performed to test the reliability of ^{14}C dating of the SOM extractions, namely soil samples from a profile in 1 excavated burial mound and samples from auguring in the cores of 9 other mounds (see Table 1).

Soil samples were collected from an excavation in the burial mound (named Hohøj) situated in Northern Jutland, Denmark. Like virtually all Danish Bronze Age burial mounds, Hohøj was built of sods only. It has a base diameter of 71 m, a height of 11.7 m, and a volume of approximately 16,630 m^3. It is one of the largest known burial mounds in northern Europe. A 10-m deep and 3-m wide excavation was opened in the western side of the burial mound to allow sampling from profiles of undisturbed recent soil processes. Samples from profile walls were collected in November 1998. Bulk soil was collected in 10 × 10 × 30 cm steel frame boxes from 2 places (Hohøj 1 and 2), 400–450 cm beneath the mound surface, respectively. Both were transported to the laboratory within 24 hr and stored at –18 °C until fractionation. Samples for fractionation were taken from the upper 0.5 to 2 cm of the surface of 2 individual sods in these boxes. The sods in the burial mound had pH (1: 1, soil:water) values of 4.4 to 4.6, and consisted of sand with 5–15% gravel and less than 5% clay. The redox status of the mound was not investigated, but redoximorphic features of Fe- and Mn-oxides were observed in a few, minor spots. Further details on the sampling and the site are given in Aaby and Andreasen (1999) and Bech (2003). As no primary burials were found in the excavation at Hohøj, no reliable archaeological dating of the construction time was possible. Two wood-sticks, identified as heather (*Calluna vulgaris*), were found in the ^{14}C-dated soil samples after wet sieving. These sticks were considered reliable controls for the ^{14}C dating of SOM, as the maximum time for visible plant remains to decompose is no more than a few decades, and as the C in sticks only resides a few decades in the living biomass before being released to the soil.

The auguring samples from barrows were obtained from 2 different groups of barrows in the southern part of Jutland, at the parishes of Tobøl and Lejrskov. In both groups, 25 to 30 barrows have been recorded with sizes varying from 10 m in diameter and 1 m in height up to 40 m in diameter and nearly 8 m in height. Today, only 9 barrows are protected by law in each group, while the remaining monuments are under cultivation and quite damaged. All barrows in the 2 groups were surveyed by auguring using a manual chamber auger with a diameter of 7 cm. In the chamber, the individual sods of the barrow construction could be distinguished as systematic sequences of layers. Sometimes, the former vegetation layer could be identified as up to 1-cm-thick, dark, organic-rich layers on top of the individual sods. Disturbances in the barrows appeared as sequences of heterogeneous fill (Breuning-Madsen and Holst, forthcoming). In the summer of 2000, samples were consistently taken from cores near the base of the barrows. Preferably, samples were from identifiable former vegetation layers. Otherwise, it was from the topmost 2 cm of a sod or the buried soil surface underneath the barrow. Subsequently, samples from 9 barrows were selected for SOM dating based on the availability of reference dating of the barrows and an evaluation of the quality of the samples, involving criteria such as the extent of disturbances in the barrow, the distinctness of the sample layer, and the certainty of the interpretation of the context of the sample. Samples were stored at –18 °C until fractionation. The dated barrows were all constructed of sandy sods with 2–8% clay and with a pH ranging from 3.4 to 4.6. The barrow Sortehøj consisted of podzolized sods with 1-cm-thick, black, humus horizons on the surfaces. In 1896, during a partial excavation of the barrow, a well-preserved oak-log coffin with grave goods from the Early Bronze Age period III (1300–1100 BC) was uncovered. The sample for the SOM dating was taken from the humus layer of a sod near the base of the barrow. The other barrows consisted of unleached or weakly-leached sods and, consequently, the sod structure was less well-defined than in Sortehøj. Strong redox features in the cores characterized the barrows Skelhøj, Sortehøj, and Lejrskov 2 and 8, where cemented iron pans had formed around an anaerobic environment in the center of the mound. In Skelhøj and Lejrskov 2, the anaerobic environment had preserved plant remains, which were used for reference dating. In the remaining 7 barrows, fragments of charcoal were gathered for maximum age (*terminus post quem*) reference dating. The charcoal was determined according to the species-level by Claus Malmros, The National Museum of Denmark.

Chemical SOM fractionation

In the organic matter fractionation procedure applied here (Dalsgaard and Odgaard 2001), the SOM was separated into 4 compartments: acid-extractable, humic acid, fulvic acid, and residual (humin). Briefly, 4–5 g of soil (with >0.1% C) was acid-washed in 50 ml 0.5% HCl, which yields the acid-extractable organic fraction (Figure 1). The soil was then treated twice with 0.5 M NaOH heated at 80 °C for 2 hr, which left a non-soluble organic fraction, the residual (humin) fraction, and a soluble fraction. Addition of 12 M HCl to the soluble fraction precipitated the humic acid fraction, whereas the supernatant contained the fulvic acid fraction. Precipitated fractions were centrifuged and all fractions freeze-dried prior to ^{14}C dating. Degassed water was used throughout and care was taken during laboratory preparation to avoid contamination from recent atmospheric CO_2. However, contaminated, modern C was probably incorporated in the humin fraction due to the high pH in this step (Hatte et al. 2001). Dating of all 4 fractions was only done in 3 samples, whereas humic and fulvic acids fractions were dated in all 10 mounds, as these fractions previously had yielded the best age estimates of burial times (Dalsgaard and Odgaard 2001). The remaining sites, where the residual fraction (which contained >90% of total SOC) was not present, represent SOM pools that are not homogenous. Yet, results from Dalsgaard and Odgaard (2001) showed using similar sandy soils, that the humic, fulvic, and residual fractions were virtually even-aged.

Table 1 Site and sampling information from the investigated burial mounds in Denmark

Site	Sb. number[a]	Location	Sample description	Sampling depth (m)
Hohøj 1	119	56°38′N; 10°00′E	Surface of sod	4.20
			surface of sod[b]	4.20
Hohøj 2	119	56°38′N; 10°00′E	Surface of sod	4.50
			Surface of sod[b]	4.50
Lejrskov 2	31	55°30′N; 9°18′E	Core of burial mound	5.10
			Humus layer on sod[b]	5.35
Lejrskov 8	2	55°31′N; 9°20′E	Buried soil surface	3.90
			Sod[b]	3.00
Skelhøj	95	55°25′N; 8°52′E	Core of burial mound	4.70
			Humus layer on sod[b]	4.00
Rishøj	61	55°25′N; 8°52′E	Sod	3.75
			Buried soil[b]	4.00
Jernved 1	56	55°25′N; 8°52′E	Buried soil	3.00
			Sod[b]	2.50
Jernved 2	57	55°25′N; 8°52′E	Buried soil surface	2.50
			Buried soil surface[b]	1.80
Sortehøj	64	55°25′N; 8°51′E	Humus layer on sod	1.65
			Oak coffin[b]	3.00
Frishøj	50	55°25′N; 8°51′E	Humus layer on soil surface	3.00
			Sod[b]	3.50
Plovshøj	49	55°25′N; 8°51′E	Humus layer on sod	2.05
			Sod[b]	2.00

[a]sb. numbers refer to a system of numbering for all Danish burial mounds
[b]reference sampling positions

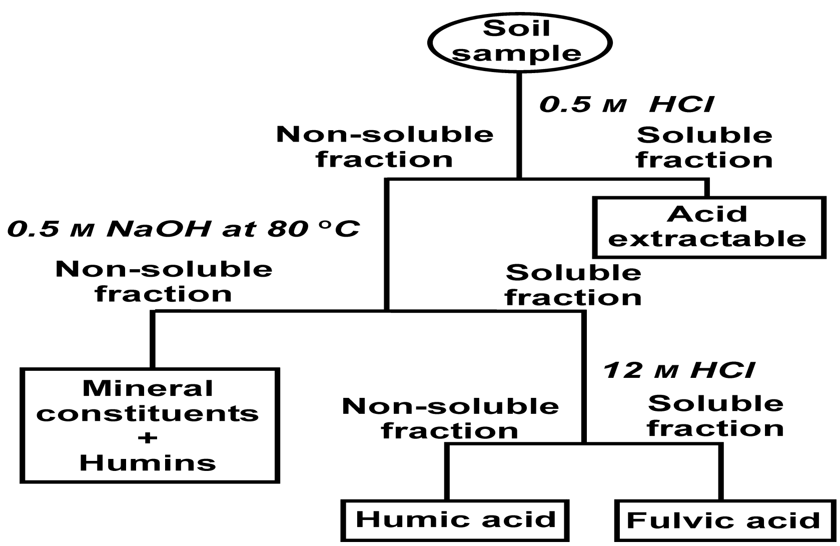

Figure 1 Chemical separation procedure for the soil organic matter fractionation. The obtained fractions are shown by boxes.

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Soil and C analysis

The heather sticks and organic C fractions were ^{14}C dated and analyzed for total C content at the AMS ^{14}C Dating Laboratory of the University of Aarhus. Calibrated ages in calendar yr were obtained from ^{14}C ages using the Seattle Program Version 3.03 (Stuiver et al. 1998). Reporting of ^{14}C data was according to Stuiver and Polach (1977). ^{14}C dating of soils are expressed by an apparent mean residence time of the SOM, which is defined as $1/k$, where k is the first-order decay constant for organic matter decomposition (e.g., Scharpenseel and Becker-Heidmann 1992).

Calculations

The usefulness of C fractionation can be evaluated by comparing the reference dating with the ^{14}C age one would obtain if the soil sample (bulk SOC) was dated without fractionation. The age of the bulk SOC can be calculated from the ages of the fractions by forming a weighted average of the ^{14}C activities, weighted with the masses of the carbon contents. The conversion of age to ^{14}C activity and back follows the expression given by Stuiver and Polach (1977).

RESULTS

The conventional ^{14}C ages of the reference samples (heather sticks) from the profile walls in Hohøj were 3180 ± 90 (±1 standard deviation) and 3085 ± 40 BP (Table 2). Heather sticks from the samples obtained by auguring revealed that Lejrskov 2 was erected around 3148 ± 37 BP and Skelhøj around 3185 ± 35 BP. The remaining mounds were reference-dated by charcoal, which was the only available macroscopic organic material. However, charcoal is known to resist microbial decomposition and its C has considerably higher apparent mean residence times compared to bulk SOC. In Lejrskov 8, Jernved 1, Frishøj, and Plovshøj, pieces of oak (*Quercus* sp.) dated to 3263 ± 46 BP, 4597 ± 46 BP, 3862 ± 49 BP, and 3391 ± 47 BP, respectively. In Rishøj, charcoal derived from alder (*Alnus* sp.) dated to 3743 ± 49 BP, and in Jernved 2, a piece from heather dated to 3415 ± 50 BP.

Dating of all fractions was only done in Hohøj 1, Lejrskov 2, and Skelhøj. The acid-extractable fraction from Hohøj had the same age (within 1 standard deviation) or was 150 yr younger than the other dated fractions. In Lejrskov 2 and Skelhøj, the acid-extractable fraction was up to 1000 yr older (Table 2). The residual fraction in Hohøj had the same age as the other fractions, and in Lejrskov 2 and Skelhøj, it was 260 and 340 yr older, respectively.

Rishøj, Jernved 1, and Frishøj did not follow the same pattern between humic and fulvic acid fractions and reference ages as the other mounds, since they had differences from 700–1520 yr. Differences between fulvic and humic acid fractions in the other 7 mounds were in contrast 460 yr or less. When excluding Rishøj, Jernved 1, and Frishøj, the fulvic acid fractions were within 1 standard deviation ($n = 4$), or were up to 460 yr older ($n = 4$) of the reference age. Again, when excluding the 3 mounds mentioned above, the humic acid fractions in the 7 other mounds were within 1 standard deviation of ($n = 4$), or up to 280 yr older ($n = 4$) than the reference ages. If excluding samples where reference ages were obtained by charcoal ($n = 5$), the fulvic and humic acid fractions were within 1 standard deviation, or were 460 and 240 yr older, respectively.

DISCUSSION

The SOM fractionation procedure

In 3 mounds—Rishøj, Jernved 1, and Frishøj—there were clearly large discrepancies (>700 yr) between fulvic, humic, and references dates (Figure 2). This suggests that combined SOM fraction

Table 2 AMS ^{14}C dating results for organic remains and extracted soil organic matter fractions from burial mounds in Denmark

Locality / Sample location	Sampling depth (m)	AMS lab number (AAR-)	Organic matter fraction	$\delta^{13}C$ (‰ VPDB)	Conventional ^{14}C age (BP)[a]	Calibrated age (cal BC)[b]
Hohøj 1	4.20	4431	Acid extractable	–24.3	2930 ± 45	1186–1128 (1256–1044)
	4.20	4430	Residual	–26.9	3075 ± 40	1375–1320 (1406–1264)
	4.20	4433	Fulvic acid	–26.9	3080 ± 40	1381–1321 (1408–1265)
	4.20	4432	Humic acid	–26.9	3145 ± 45	1413 (1487–1324)
	4.20	4288	*Calluna vulgaris* stick	–27.4	3180 ± 90	1440 (1520–1320)
Hohøj 2	4.50	4434	Humic acid	–26.9	3160 ± 50	1429 (1494–1399)
	4.50	4289	*Calluna vulgaris* stick	–27.6	3085 ± 40	1386–1322 (1409–1310)
Lejrskov 2	5.10	7327	Acid extractable	–27[c]	3770 ± 80	2200–2150 (2295–2040)
	5.10	7328	Residual	–26.84	3401 ± 45	1726–1689 (1742–1637)
	5.10	7329	Fulvic acid	–26.31	3421 ± 45	1737–1693 (1766–1644)
	5.10	7330	Humic acid	–27.36	3362 ± 48	1681–1640 (1735–1535)
	5.5	7345	*Calluna vulgaris* stick	–28.18	3148 ± 37	1425–1414 (1485–1399)
Lejrskov 8	3.90	7343	Fulvic acid	–26.24	3715 ± 60	2135–2065 (2200–1985)
	3.90	7344	Humic acid	–26.41	3545 ± 39	1883–1835 (1936–1779)
	3.00	7553	*Quercus* sp. charcoal	–24.14	3263 ± 46	1521 (1603–1462)
Skelhøj	4.70	7331	Acid extractable	–27[c]	4215 ± 75	2880 (2900–2680)
	4.70	7332	Residual	–26.98	3521 ± 40	1879–1829 (1890–1770)
	4.70	7333	Fulvic acid	–26.41	3645 ± 47	2026–1981 (2126–1941)
	4.70	7334	Humic acid	–27.29	3422 ± 43	1737–1693 (1766–1664)
	4.00	7346	*Calluna vulgaris* stick	–28.78	3185 ± 35	1438 (1504–1413)

[a]The ^{14}C ages are given in conventional radiocarbon yr BP (before present = 1950), with a measuring uncertainty of 1 standard deviation.
[b]Calibrated ages in calendar yr (Stuiver et al. 1998). The first numbers represent the range of intercepts with the calibration curve. The age range in parentheses corresponds to the ±1 standard deviation in the conventional ^{14}C age.
[c]Standard value assumed

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Table 2 AMS ^{14}C dating results for organic remains and extracted soil organic matter fractions from burial mounds in Denmark *(Continued)*

Locality / Sample location	Sampling depth (m)	AMS lab number (AAR-)	Organic matter fraction	$\delta^{13}C$ (‰ VPDB)	Conventional ^{14}C age (BP)[a]	Calibrated age (cal BC)[b]
Rishøj	3.75	7323	Fulvic acid	–27.06	2320 ± 55	395 (405–265)
	3.75	7324	Humic acid	–27[c]	3290 ± 120	1600–1530 (1740–1430)
	4.0	7554	*Alnus* sp. charcoal	–27.38	3743 ± 49	2186–2141 (2266–2040)
Jernved 1	3.00	7339	Fulvic acid	–26.32	3356 ± 39	1680–1638 (1688–1540)
	3.00	7340	Humic acid	–27[c]	3080 ± 80	1380–1320 (1430–1220)
	2.50	7556	*Quercus* sp. charcoal	–26.81	4597 ± 46	3362 (3492–3348)
Jernved 2	2.50	7335	Fulvic acid	–26.69	3521 ± 50	1879–1829 (1917–1750)
	2.50	7336	Humic acid	–27.10	3400 ± 65	1725–1690 (1765–1620)
	2.50	7557	*Calluna vulgaris* charcoal	–26.45	3415 ± 50	1736–1692 (1766–1640)
Sortehøj	1.65	7326	Fulvic acid	–28.12	2976 ± 43	1256–1133 (1290–1128)
	1.65	7325	Humic acid	–28.13	3017 ± 43	1288–1262 (1372–1133)
	3.00	—	Oak coffin	—	—	(1300–1100)[d]
Frishøj	3.00	7337	Fulvic acid	–26.51	3824 ± 49	2285–2213 (2397–2153)
	3.00	7338	Humic acid	–27[c]	3170 ± 110	1430 (1520–1320)
	3.50	7555	*Quercus* sp. charcoal	–23.59	3862 ± 49	2326–2307 (2458–2207)
Plovshøj	2.05	7341	Fulvic acid	–26.35	3678 ± 48	2110–2035 (2138–1977)
	2.05	7342	Humic acid	–27.06	3669 ± 42	2033 (2136–1977)
	2.00	7558	*Quercus* sp. charcoal	–23.53	3391 ± 47	1687 (1741–1623)

[a]The ^{14}C ages are given in conventional radiocarbon yr BP (before present = 1950), with a measuring uncertainty of 1 standard deviation.
[b]Calibrated ages in calendar yr (Stuiver et al. 1998). The first numbers represent the range of intercepts with the calibration curve. The age range in parentheses corresponds to the ±1 standard deviation in the conventional ^{14}C age.
[c]Standard value assumed
[d]Dated by grave goods

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and ^{14}C analysis yielded an estimate of the reliability of the ^{14}C age, as contamination of SOM or reference sample problems were reflected by age discrepancies.

The acid-extractable fraction in these acid soils is suggested to extract young, dissolvable SOC (Wang and Hseih 2002). Younger C may have contaminated the acid-extractable fraction from Hohøj, as this fraction was up to 150 yr younger than the other dated fractions (Table 2). The fractions were probably caused by water-soluble C leached from the mound surface after its construction. In contrast, the acid-extractable fractions from Lejrskov 2 and Skelhøj were up to 1000 yr older than the other dated fractions. This signifies a considerable contamination with older SOC, either derived from water-soluble C in the mound or as the SOC were not homogeneous pools before the sods were placed in the mound. The acid extraction elucidated and, to some degree, probably eliminated, contamination of the remaining fractions originating from water-soluble C.

The residual fraction (humin) is a poorly-known, but highly refractory, pool of SOM (Rice 2001) consisting of insoluble organic remains, e.g. pollen. The residual fraction from the 3 dated samples did not differ from the humic and fulvic acid fractions by more than 250 yr. This signifies that the SOM from the sod surfaces were probably derived from an ecosystem where C was rejuvenated with a considerable speed (i.e., as expected when sampling former soil-surface horizons).

The fulvic acid fraction is believed to be a broad group of organic acids, which are mobile in the upper soil during soil formation, e.g. podzolization. Accordingly, although both the fulvic and humic acid fractions had good correlation with references ages, the humic acid ages were probably the most reliable, since the mobility of humic acids in soil is low and the humic acid fraction generally gives the most precise dates of burial time for soil surfaces (van Mourik et al*.* 1999; Dalsgaard and Odgaard 2001). This accordance between humic and fulvic acid fractions and reference ages was probably related to dynamics turnover times of the different pools of organic C in the original Bronze Age soil. Thus, the humic acid age should be corrected for its apparent mean residence time, which presumably was 280 yr or less. The uncertainty on the time of burial, thus, depended on ^{14}C dating accuracy, the organic matter's age before incorporation in the SOM, and the mean residence time of the SOM. The latter especially varies greatly between soil types, land uses, vegetation types, climates, etc. (e.g., Wang et al. 1996). The uncertainty introduced by these 280 yr increased the total uncertainty of the archaeological dating considerably but the obtained ages still allow burial mounds to be grouped into archaeological periods, as they cover several centuries.

If considering the fractionated samples as 1 bulk sample of the whole soil, the usefulness of the SOM fractionation could be evaluated with respect to ^{14}C dating. For example, we can examine the reference dates obtained from Skelhøj (3185 ± 35 BP) with the humic acid fraction being approximately 250 yr older (Table 2). Calculations on an apparent mean residence time of bulk SOC, as one would obtain if the soil sample was dated without fractionation, gave an age estimate of 3477 BP. In Lejrskov 2, the (calculated) age of bulk SOC (3382 BP) would not have departed (within 1 standard deviation) from the age obtained by the humic acid fraction (3362 ± 48 BP). However, although the humic acid fraction in Skelhøj overestimated the age by 250 yr, a ^{14}C dating without fractionation would give an additional increase of 55 yr in the estimated age. In addition, without fractionation there would be no indication of the uncertainty or contamination in either of the mounds, since apparent mean residence time of bulk SOM yields no information on this. The ^{14}C age without SOM fractionation would (in all our samples) approach the age of the residual fraction, since it contained 1–2% C, but weighed around 100 times more than the other fractions all together; it contained >90% of the SOC. Hence, SOM fractionation seemed important for both dating reliability and age estimates.

The visibility of the former vegetation layer or soil surface seemed highly important for precise dating. In the Hohøj and Sortehøj mounds, where the upper 1 cm or less of the former vegetation/humus layer was sampled only, ages of humic acids and references were within 1 standard deviation (Tables 1 and 2). Where the surfaces of the former soil or sod were less clear, as in Lejrskov 2 and 8, Skelhøj, and Plovshøj, the age discrepancy between humic acids and references was up to 280 yr. A precise sampling of an organic-rich, former surface layer, thus, yielded the most reliable samples for SOM fractionation. Such surface layers were probably exclusively built-up of plant remains that were not mixed with C from the subsurface SOC, i.e. on a surface that was not ploughed for decades.

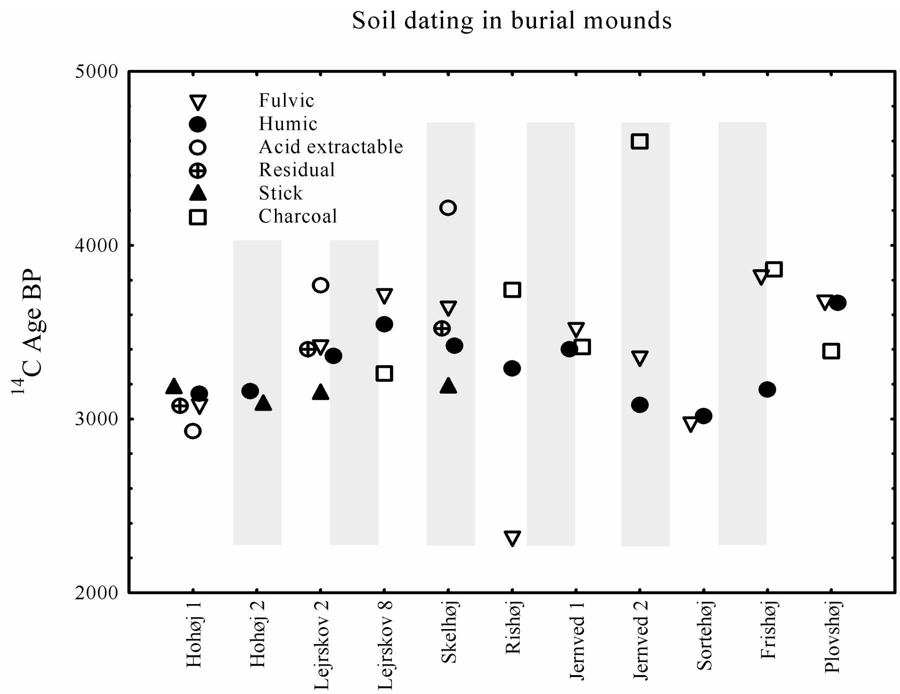

Figure 2 AMS ^{14}C dating results for organic remains and extracted soil organic matter fractions from burial mounds in Denmark. Standard deviations are in Table 2. Note that error bars are omitted, as they are smaller, in most cases, than the symbols.

Problems with ^{14}C dating SOM

Problems associated with ^{14}C dating of SOM in burial mounds are the following:

- Organic matter near the surface can be relocated by physical and chemical processes;
- Certain SOM fractions decompose slowly;
- SOC, which originates from above and below ground plant parts, can be recycled by soil biota.

The sum of these processes will make organic C in buried soil surfaces appear older than the actual time of burial (e.g., Wang et al. 1996). However, this will cause an error of less than a century (at maximum) when sampling the upper 0.5–2 cm of the former soil surface or humus layer from most ecosystems (Bol et al. 1999; Gaudinski et al. 2000). Furthermore, as the buried soil underneath the burial mounds often show evidence of ploughing by a primitive plough (an ard), sampling should also be restricted to the uppermost 1–2 cm here because tillage may increase SOM ages by several centuries (Paul et al. 1997). A careful sampling of the few cm previously at the soil surface is accordingly very important, as our good results from the well-defined surface layer in Sortehøj and Hohøj emphasized. Channels and pores formed by macrofauna and roots should also be avoided, if possible, as they may contain younger C. Soil mixing is often highly localized (Grave and Kealhofer 1999), and hence, can be avoided in profiles below a certain depth. This is typically 0.5–1 m in Danish burial mounds. For samples taken nearer to the present-day surface than the minimum depth of 1.6 m, here the problem of younger C and bioturbation becomes increasingly important, as bioturbation, biological production, and C turnover increase considerably above this depth (e.g. Bird et al. 2002; Gaudinski et al. 2000).

Physical transport of the SOM is commonly found in many soils and burial mounds as clay-humus illuviation bands that can be confounded with buried surfaces. A careful macroscopic examination, however, revealed that the upper boundary of the illuviation band was abrupt (a few mm), whereas the upper boundary of sods was more gradual. Thus, erroneous sampling can be minimized and verification can be done by a soil-thin section description. With careful and precise sampling, contamination can be restricted to chemical transport of dissolved organic C and SOC present in the soil before burial. The problem of contamination of younger, water-soluble C, or older SOC present in the soil before burial, can be evaluated by the fractionation procedure applied here, since discrepancies larger than a few decades between the residual, fulvic, and humic fractions apparently signify that the SOC originally was not a homogenous pool. A more advanced SOM fractionation can, in theory, circumvent these problems. This could be done in the following ways: i) by ^{14}C dating of aliphatic hydrocarbons in the SOM, which have low mobility in soils and are formed by plant leaves (Huang et al. 1999); ii) physical fractionation by ^{14}C dating of particulate organic matter (250 to 2000 μm), which have very low apparent mean residence times (e.g. Bird et al. 2002); or iii) by a combination of both.

Archaeological results from the burial mounds

Pollen analysis from Hohøj revealed a pollen assemblage typical for Danish burial mounds from the Bronze Age (Aaby and Andreasen 1999). This accords with the ages of the 2 heather sticks, the humic acid, fulvic acid, and residual fractions, all of which give dates around 1400 BC (Table 2; Figure 2). Hohøj was accordingly erected in the Early Bronze Age, probably shortly before 1400 BC. When the total uncertainty is considered, this corresponds to the Late Period II. Thus, Hohøj was approximately 500 yr older than burial mounds of comparable size elsewhere in Scandinavia; although remains of a Swedish burial mound with a similar diameter, but unknown original height, also dates to 1500–1300 BC (Randsborg 1993).

The 9 burial mounds explored by soil auguring and buried soil surfaces could not be sampled as precisely as in the case of Hohøj, where a soil profile was available. Nevertheless, dating where reference dates and fulvic and humic fractions were within an interval of <280 yr was established for Lejrskov 2, Skelhøj, Jernved 2, and Sortehøj. Lejrskov 2 was erected about 1414–1425 BC; Skelhøj was erected around 1438 BC; Jernved 2 most probably was constructed from about 1738–1692 BC; and Sortehøj was correspondingly erected around 1288–1262 BC. More uncertainty exists in age estimates of Lejrskov 8 and Plovshøj, as they had high age discrepancies between fulvic and humic fractions. Yet, Lejrskov 8 was probably constructed around 1600–1450 BC and Plovshøj around 1750–1600 BC, as the reference age and humic acids (–280 yr) signified. The charcoal found in the burial mounds at Jernved 1 and Rishøj was believed to pre-date the burial mounds' construction, as they were, respectively, 1240–1520 and 450–1400 yr older than the SOM fractions. This may apply to

Frishøj as well because of the 700 yr discrepancy between humic and fulvic acid fractions. Accordingly, Rishøj was probably constructed around 1600–1300 BC, whereas both Jernved 1 and Frishøj were constructed around 1400–1000 BC. This emphasizes that charcoal as a reference age needs to be interpreted cautiously and seen as a *terminus post quem* age only, but also that the combination with SOM-fraction dating gives an estimate of the reliability of the charcoal age, or *vice versa*.

All dates fall within the Early Bronze Age (1700–1000 BC) and apparently with a concentration in Period II (1500–1300 BC). It is a relatively narrow timespan compared to the 2-millennia-long timespan from the early 3rd millennium BC to the early 1st millennium BC, where barrows were constructed in large numbers in south Scandinavia. This pattern is interesting in relation to the discussion on how the distinct barrow groups came into existence, since prehistoric barrows often appear concentrated in distinct groups across the landscape in Denmark. From previous excavations, it appears that these barrow groups are characterized by mounds with particularly complex constructions and unusually lavish Bronze Age grave goods. These characteristics are strong indications of areas with a special role in the prehistoric society.

The problem with respect to archaeological interpretation of ^{14}C dating is especially bioturbation (e.g., by earthworms), which mixes the soil horizons either before or after burial. Our results and general soil science knowledge suggest that the distinctness of the buried soil layer reflects the SOC dynamics in most soils. A visual inspection of the soil morphology in the profile walls will, thus, in most cases, *a priori* determine the usefulness of a ^{14}C dating. A soil with low macrofauna activity, as are common in acidic environments and cold or dry climates, thus creates the best conditions for ^{14}C dating of SOM fractions.

CONCLUSIONS

Our results showed that even auguring by a hand-operated soil augur gives suitable samples from former surface soils for AMS ^{14}C dating of SOC, though the certainty of the sample's context inevitably will be higher in excavations. One advantage of this procedure was that the reliability of ^{14}C dating was already indicated in the field by the distinctness of buried organic-containing layers. The disturbance of the present-day soil surface was minimal and sampling did not change the mounds' redox status, which in some cases have preserved organic grave goods and corpses for millennia. As many of the northern European burial mounds were constructed from numerous layers of sods, or were constructed above a former soil surface, we conclude that ^{14}C dating of SOM fractions has the potential of being a useful tool in archaeological investigations. However, to achieve a method for archaeological dating that can be applied worldwide, a more advanced fractionation of the SOM is probably required.

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