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Ojala, Antti

2019

Ojala , A , Saarnisto , M , Jungner , H , Snowball , I & Muscheler , R 2019 , ' Biases in radiocarbon dating of organic fractions in sediments from meromictic and seasonally hypoxic lakes ' , Bulletin of the Geological Society of Finland , vol. 91 , no. 2 , pp. 221-235 . https://doi.org/10.17741/bgsf/91.2

http://hdl.handle.net/10138/308675
https://doi.org/10.17741/bgsf/91.2.004

Bulletin of the Geological Society of Finland, Vol. 91, 2019, pp 221–235 https://doi.org/10.17741/bgsf/91.2.004

Biases in radiocarbon dating of organic fractions in sediments from meromictic and seasonally hypoxic lakes

Antti E. K. Ojala[1]*, Matti Saarnisto[2], Högne Jungner[3],
Ian Snowball[4] and Raimund Muscheler[5]

[1] *Geological Survey of Finland, Vuorimiehentie 5, FIN-02151, Espoo, Finland*

[2] *Mikonkatu 22 D 46, FIN-00100 Helsinki, Finland*

[3] *University of Helsinki, PO Box 64, FIN-00014 Helsinki, Finland*

[4] *Department of Earth Sciences, Uppsala University, Villavägen 16, 752 36 Uppsala, Sweden*

[5] *Department of Geology, Lund University, Sölvegatan 12, SE-22362 Lund Sweden*

Abstract

We present here radiocarbon dating results from two boreal lakes in Finland, which are permanently (meromictic) or seasonally stratified and contain continuous sequences of annually laminated sediments that started to form in the early Holocene. The radiocarbon dating results of different organic components were compared with the varve-based sediment chronologies. The deviation between the Lake Valkiajärvi varve chronology (8400 varve years ±2–3% error estimate) and 33 ^{14}C dates taken from insoluble and soluble organic phases vary inconsistently throughout the Holocene. In extreme cases mean calibrated radiocarbon dates with 95.4% confidence levels (2σ) are -2350 and +2040 years offset when compared with the varve chronology. On average, the radiocarbon dates are offset by ca. +550 years. The deviation between the Lake Nautajärvi varve chronology (9898 varve years ±1% error estimate) and 26 ^{14}C dates analyzed with conventional and AMS methods indicates that radiocarbon dates are systematically older by 500–1300 years (about 900 years on average). This significant offset mean that radiocarbon dates obtained from organic bulk sediment of meromictic and seasonally hypoxic lakes must be cautiously interpreted because of the reservoir effect and carbon cycling at the sediment-water interface. Direct evidence was obtained from the dating of soluble fraction and insoluble organic matter from near bottom water in the monimolimnion of Lake Valkiajärvi, which yielded ^{14}C ages of 560 ±80 BP and 2070 ±140 BP, respectively. Our study reinforces previous results that age-depth models based on bulk sediment radiocarbon dates obtained on sediments of stratified lakes are of limited value for accurate dating of changes in land use and especially the commence of agriculture.

Keywords: lake sediment, dating, radiocarbon, varves, anoxia, Finland

*Corresponding author: (e-mail: antti.ojala@gtk.fi)

Editorial handling: Juha Pekka Lunkka (e-mail: juha.pekka.lunkka@oulu.fi)

1. Introduction

Lake sediments are commonly radiocarbon dated. In the conventional technique, due to sample size requirements, bulk sediment samples were often used and the same practice continued to some extent in paleo-ecological studies when the AMS-technique was introduced, although adequate amounts of carbon can often be received by preparing single terrestrial plant macro-fossils such as seeds or needles from sediment cores (e.g. Fowler et al., 1986; Tuniz et al., 2004). Dating results of bulk sediment samples may be erroneous because the sample contains organic components of different age. Varved sediments, on the other hand, provide a possibility to compare radiocarbon dating results of different methods and different organic components with the inherent varve chronology that is based on counting of seasonal layers (Zolitschka et al., 2015).

Several deep small lakes in the Lammi area south Finland are kettle holes in eskers. Many of them are meromictic and their sediments are annually laminated. Their value as archives of past environmental change was recognized in several studies in the 1970´s (Kukkonen & Tynni, 1972; Saarnisto et al., 1977; O'Sullivan, 1983; Wetzel, 1983) and their discovery has led to a number of paleo-ecological studies on the history of vegetation and agriculture (e.g. Tolonen, 1978a, 1978b; Tolonen, 1980; Huttunen, 1980) as well as methodological approaches for understanding of varve formation (e.g. Simola, 1979). The meromixis in lakes became known in Finland due to the study of Lake Valkiajärvi in Ruovesi by Meriläinen (1970), and in tests done of the freezing method developed by H.E. Wright Jr (Wright, 1980) and introduced to Finland 1972 (Saarnisto, 1975; 1986) the sediments were confirmed to be annually laminated.

It became soon evident in studies of annually laminated lake sediments in the Lammi area that ages obtained by varve counting and radiocarbon deviated considerably, which cannot be explained by physical mixing of the sediments because of absence of bioturbation. This discrepancy was observed in several cases; in Lake Ahvenainen as discussed by Tolonen (1978b), in Lake Valkiajärvi (Saarnisto, 1987), in Lake Lampellonjärvi (Tolonen, 1980), and in Lake Lovojärvi (Saarnisto, et al. 1977; Huttunen & Tolonen, 1977 and Huttunen, 1980). All these studies concluded that reworking of old carbon from cleared meadows and agricultural fields in the catchment of lakes resulted in bulk sediment dates several hundreds of years too old in comparison with the sediment varve counts. In the case of Lovojärvi, for example, the discrepancy varied between 720 and 980 years. Increased varve thickness in upper sediment sequences indicates increased erosion in the lake catchments due to anthropogenic activities (Saarnisto et al., 1977). Another source of error was the varve count, which was mostly undertaken from frozen surface of cores. Difficulties were pronounced in pre-culture organic sediments where annual sediment accumulation was only 0.2 to 0.3 mm, as acknowledged by the authors.

This study discusses the still underappreciated problems with radiocarbon dates from bulk sediments of permanently or seasonally hypoxic lakes. We provide a word of caution regarding the use of bulk radiocarbon dates from lakes of permanent (or seasonal) stratification using

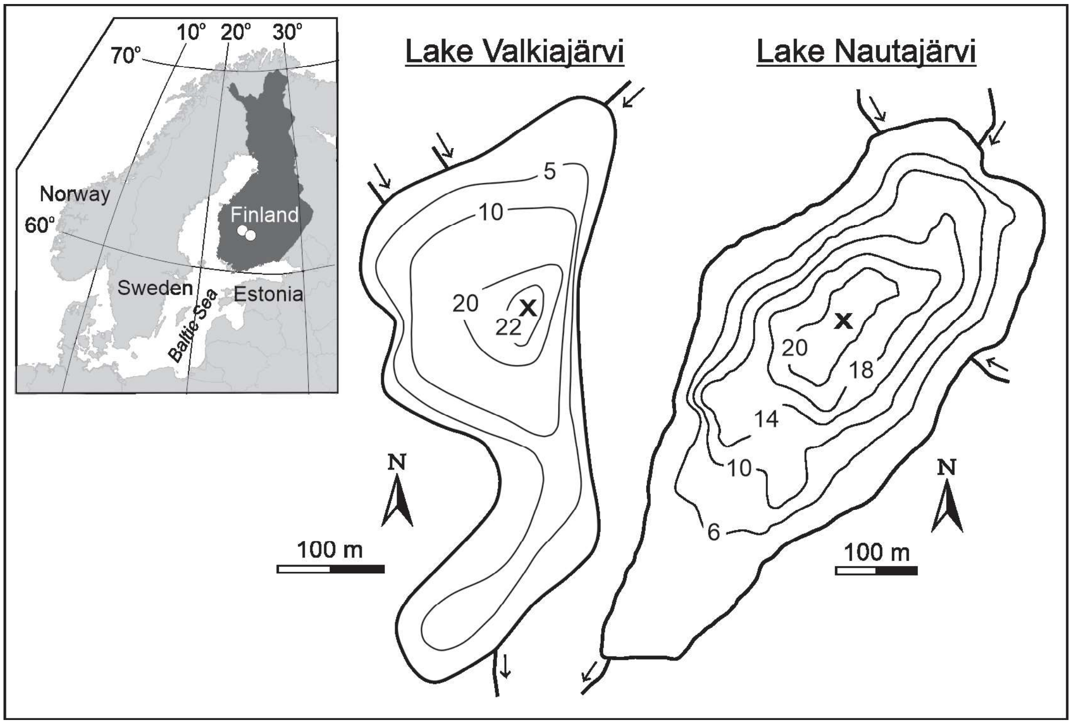

Figure 1. Location and bathymetry of lakes Valkiajärvi and Nautajärvi in southern central Finland (white circles in the inset map). The sediment cores analysed for radiocarbon were taken from the deepest points, which are marked with a black cross in each case. Detailed description of stratigraphical units from sediment sections are given in Saarnisto (1985), Ojala and Saarnisto (1999), and Ojala and Alenius (2005).

two examples from Lake Valkiajärvi and Lake Nautajärvi. We compare dating results between radiocarbon analysis and varve counting, and discuss of reasons for their deviation.

2. Materials and methods

Lake Valkiajärvi is located in the Fennoscandian boreal forest zone in central-southern Finland (61°54'N, 23°53'E, 110 m a.s.l.) (Fig. 1). The lake has an area of 8 ha and a maximum depth of 26 m within a single basin. The bedrock consists of granites and quartz diorites that are partly overlain by till and the lake is surrounded by hills that rise to 150–160 m a.s.l. The lake is well sheltered from wind and the small catchment area is uninhabited by humans and dominated by coniferous trees. Valkiajärvi is an oligotrophic and meromictic lake with permanent chemical stratification (Meriläinen, 1970; Ojala & Saarnisto, 1999). The chemocline lies at the depth of 17 m and the mixolimnion above that covers about 93% of the water mass and undergoes spring and autumn overturns (Meriläinen, 1970). The monimolimnion is rich in dissolved substances, such as Fe, Ca, Na, and contains large amounts of dissolved gases, and is anoxic. The water of the monimolimnion is also dark in colour indicating high concentration of organic compounds (Meriläinen, 1970).

Lake Nautajärvi is located in central southern Finland (61°48'N, 24°41'E, 103.7 m a.s.l.) (Fig. 1). The lake is oval-shaped and covers an area of 17 ha and has a maximum depth 20 m within a single basin (Ojala and Alenius, 2005). Deeply eroded coarse-grained granites, partly overlain by a thin till layer, cover approximately half of the drainage basin, while the other half is covered by thicker superficial deposit of glacial till and postglacial silt in equal proportions. Like Valkiajärvi, Lake Nautajärvi is well sheltered from wind and the catchment area is dominated by pine (*Pinus sylvestris*) and spruce (*Picea abies*). Lake Nautajärvi is dimictic with seasonal water stratification and anoxia near the bottom water (Ojala et al. 2013). Water conductivity varies between 3.1 and 3.4 MS M^{-1} and pH (5.8–6.0) is fairly stable throughout the water column. Concentrations of Fe and Mn increase with depth from 500 to 2000 $\mu g\ l^{-1}$ and from 100 to 500 $\mu g\ l^{-1}$, respectively, during seasonal stratification. Total N and P in the water column are also highest near the bottom and vary between 400 and 800 $\mu g\ l^{-1}$ and between 15 and 80 $\mu g\ l^{-1}$, respectively, indicating mesotrophy (Ojala and Alenius, 2005).

2.1. Sediment samples, stratigraphy and varve counts

Lakes Nautajärvi and Valkiajärvi preserve clastic-organic varves (e.g. Ojala et al., 2012; Zolitschka et al., 2015). Their sedimentation reflects an annual cycle of catchment erosion (minerogenic matter) and influx during spring floods, accumulation of particulate organic material (e.g. plants, algae) in summer to autumn, and deposition of a thin fine-grained and dark-colored organic layer in winter that is deposited under the ice-cover (Renberg, 1983; Ojala et al., 2000). The deposition environment and detailed physical properties of this type of varves have been presented in Tiljander et al. (2002), Ojala and Francus (2002) and Ojala and Tiljander (2003). The varved sediments provide a basis for high-resolution studies and the possibility to calculate seasonal rates of sediment increments as the sediments do not undergo mixing with younger or older material. Most importantly, varved sediments provide an inherent and continuous timescale – the varve chronology – for paleoclimate studies and for evaluation of other dating methods (Ojala et al., 2012; Zolitschka et al., 2015).

The sediment of Lake Valkiajärvi was sampled on several occasions in 1975–1977 and again in 1996 by using in situ freezing method and various types of piston cores up to 90 mm sample tube diameter. The coring methods and available cores are described in Ojala & Saarnisto (1999). In addition to sediment coring, a water sample of several tens of liters was carefully collected from near the bottom at the depth of 24 m for radiocarbon analysis. The collected water was dark brown in colour and gas was released when the sample was raised to the surface of the lake ice and the pressure eased.

The sediment succession in the center of Lake Valkiajärvi represents late- to postglacial clay overlain by a 330 cm thick section of Holocene clastic-biogenic lacustrine varves (Saarnisto, 1985; Ojala & Saarnisto, 1999). Because of a rather slow rate of sediment accumulation (average of 0.3 mm a^{-1}) in Lake Valkiajärvi, the chronological error in the varve chronology was based on repeated varve counts by different authors and ten parallel sediment sequences. The established varve chronology was found to cover 8400 varve years with the estimated chronological error of 2–3% (Ojala & Saarnisto, 1999). This margin of error is typical for varved sediment chronologies worldwide (Ojala et al. 2012). We note that in the Valkiajärvi section, the majority of dating uncertainty accumulates within the depths of 24–65 cm, representing ca. 600–1400 cal. BP. In the present paper, we use the updated varve count labeled as "new count by A.O." in the Ojala and Saarnisto (1999) study.

Immigration of *Picea* to the Valkiajärvi area is a valuable tool to check the varve chronology. In the pollen analysis from the sediments of Valkiajärvi *Picea* (spruce) pollen percentages increased from less than 1 to 6% within 70 years approximately 4400 varve years ago and the annual pollen influx values from less than 200 to nearly 800 pollen grains/cm^2 indicating the immigration of spruce into the area, which is in good agreement with the calibrated radiocarbon age for that event in the region (Aartolahti, 1966; Saarnisto, 1987; Ojala & Saarnisto, 1999).

Lake Nautajärvi was cored several times during 1999–2015 and more than 20 continuous

7–8-m-long sediment sequences were recovered (e.g. Ojala and Tiljander, 2003; Ojala and Alenius, 2005). Lake Nautajärvi varves were counted using X-ray densitometry coupled with digital image analysis and semi-automated counting of annual/seasonal layers (Ojala, 2004). Varve counts were repeated 2–5 times by two authors on three separate cores in order to establish the final varve chronology (Ojala and Tiljander, 2003). The possible extra varves (+ error) and missing varves (- error) identified during repeated varve counts on different sections were included in the varve chronology as an estimate of the quality and included in the age scale as the maximum chronological error. Lake Nautajärvi varves extend continuously from the present day to 9852 cal BP (9898 varves). The fidelity of the Lake Nautajärvi sediment chronology is discussed in detail by Ojala and Tiljander (2003), and it is estimated to be reliable within ±1% margin of error. The varve chronology was also cross-checked by comparing palaeomagnetic measurements with other independently varve-dated palaeomagnetic records from Finland (Ojala and Tiljander, 2003) and Sweden (Snowball and Sandgren, 2002; Zillén et al., 2003).

2.2. Radiocarbon dating

A number of radiocarbon samples from lakes Nautajärvi and Valkiajärvi sediment sequences were dated in three laboratories, the Radiocarbon Dating Laboratory of the University of Helsinki, Finland (Jungner 1979), the Radiocarbon laboratory at the Geological Survey of Finland (GTK) (not operational anymore), and the Radiocarbon Dating Laboratory at Lund University, Sweden (Skog et al., 2010).

With Valkiajärvi, the original idea in the 1970's was that varved sediments from non-calcareous sediments of soft water lakes offer a tool for radiocarbon/calendar year comparison (Stuiver, 1969). A total of 35 samples for conventional radiocarbon age determination were taken from cores VAL-75, VAL-76, VAL-77 and one sample of organic matter from a water layer close to the lake bottom (Table 1). Samples for radiocarbon dates were taken from several cores that were correlated with distinct marker layers within varved sequence as described by Ojala & Saarnisto (1999). For discussion and comparison with the Valkiajärvi varve chronology, we concentrate here mainly on ^{14}C analysis of samples from the core VAL-77, in which insoluble (INS) and soluble (SOL) phases were determined from the same sediment depth levels and analyzed as consecutive samples (Table 1). On the basis of the varve counts, these seven samples consisted of a total of 200 varves that were taken at 1000 varve-year intervals between 1000 and 7000 cal. BP. After the first standard AAA treatment, both the fraction insoluble (INS) in sodium hydroxide and the soluble (SOL) humus fraction were dated. That applied also to the large bottom-near water samples.

With regards to Nautajärvi, a total of 26 ^{14}C samples were analysed. The first 15 samples, each 10-cm-thick and covering about 100–200 years of sedimentation, were distributed evenly throughout the Holocene and taken as bulk samples for conventional dating at GTK. These analyses were supplemented with 11 AMS ^{14}C dates of bulk sediment around the 8.2 ka (cooling) event (Alley et al., 2005; Veski et al., 2004), which were analysed at Lund University. These samples were taken at 10-years-resolution (0.5–1.0 cm) using varve counting as guidance.

We used a Bayesian P-sequence deposition model in Oxcal 4.3 software by Bronk Ramsey (2008, 2009) to calibrate the radiocarbon dates and to provide the Bayesian sediment age-depth model for different components (humic, SOL, and insoluble fraction of bulk gyttja, INS) of organic remains.

3. Results

The deviation between varve counts and ^{14}C dates from different components (gyttja INS and humic SOL) vary inconsistently in the whole ^{14}C data from various frozen and unfrozen cores of the sediments

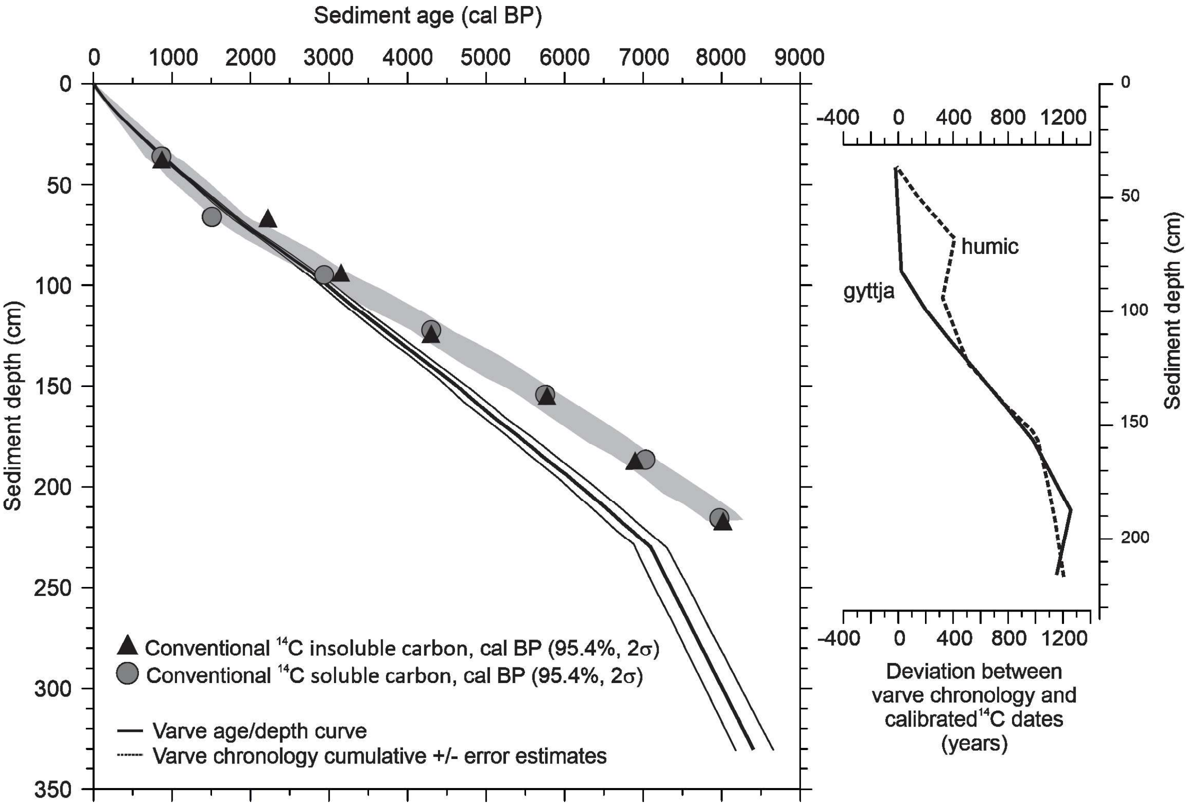

Figure 2. Varve counting-based age-depth model for Lake Valkiajärvi sequence with ±3% error estimates (see Ojala and Saarnisto, 1999). Calibrated radiocarbon mean ages for INSOL (circles) and SOL (triangle) components of core VAL-77, and Bayesian P-sequence (shaded gray) for gyttja-based dates. The graph on the right hand side shows the deviation between the varve chronology and the calibrated ^{14}C dates from different components.

of Lake Valkiajärvi (Table 1). The most extreme offsets between varve age (Ojala & Saarnisto, 1999) and calibrated radiocarbon dates are -2030 and +2620 years. On average, the dates are offset by ca. 550 years. In the following section, the deviation between the varve chronology and a set of ^{14}C dates from the longest VAL-77 core is presented. Radiocarbon dates that are 'too young' compared to varve ages occasionally appearing in the upper part of the Lake Valkiajärvi sequence are all from the same VAL-76 core. These may be related to unclean sampling during core recovery and/or subsampling, or errors in core correlation.

Figure 2 illustrates the deviation between the varve chronology and calibrated ^{14}C dates from different components, i.e. gyttja (INS) and humic (SOL), in core VAL-77. The deviation increases towards the older dates from ca. -100 years at 1000 varve years ago to ca. +1200 years at 7000 varve years ago. The ^{14}C age of the INS gyttja component at 2000 varve years ago was 350 years older and that of the SOL humic component 400 years younger than the varve age. Otherwise, both dated components gave closely similar ages. The reservoir effect in the Lake Valkiajärvi sediment sequence thus varies between -350 and +1250 years.

The deviation between ^{14}C dates and varve counts in the Lake Nautajärvi section is different to the Lake Valkiajärvi case as calibrated radiocarbon dates are systematically 500–1600 years older than the dates based on a varve chronology (Ojala and Tiljander, 2003) (Table 2). It is noteworthy that this deviation is independent of the sampling resolution and radiocarbon dating method (conventional

Table 1. Conventional radiocarbon dates from the sediment samples of Lake Valkiajärvi and from the bottom near (below 24 m) water of the deepest part (24.5 m) of the lake. Presented are also the varve ages based on Ojala and Saarnisto (1999) and the deviations between calibrated ^{14}C dates and the varve chronology.

Sample	Type	Lab ID	Depth (m)	^{14}C age BP	^{14}C age ±	^{14}C cal BP (95.4%, 2s)	Most likely ^{14}C age (cal BP)	Varve age (cal BP)	Age deviation (yrs)
	water (gas)	Hel-1917	24	560	80	783-675	583	N/A	N/A
	water (org)	Hel-1973	24	2070	140	1714-2349	2051	N/A	N/A
VAL-75	gyttja/mud	Hel-194	1.25 - 1.40	4040	210	3896-5053	4560	4050	-154 – +1003
VAL-75	gyttja/mud	Hel-195	1.75 - 1.90	6320	160	6850-7512	7230	6150	+700 – +1362
VAL-75	gyttja/mud	Hel-196	2.45 - 2.60	7970	180	8413-9304	8810	7150	+1263 – +2154
VAL-75	gyttja/mud	Hel-982	1.15 - 1.20	5540	180	5928-6698	6370	4480	+1448 – +2218
VAL-75	gyttja/mud	Hel-983	1.60 - 1.65	6850	210	7323-8054	7720	5100	+2223 – +2954
VAL-75	gyttja/mud	Hel-1021	2.51 - 2.61	8350	170	8952-9691	9320	7350	+1602 – +2341
VAL-75	gyttja/mud	Hel-1023	2.085 - 2.185	7780	160	8307-9021	8590	6470	+1837 – +2551
VAL-76	gyttja/mud	Hel-1022	0.24 - 0.34	690	110	508-800	630	1090	-582 – -290
VAL-76	gyttja/mud	Hel-1024	1.135 - 1.193	3050	140	2878-3560	3230	3540	-662 – +20
VAL-76	gyttja/mud	Hel-1114	0.645 - 0.745	1980	90	1709-2152	1930	2350	-641 – -198
VAL-76	gyttja/mud	Hel-1115	0.85 - 0.95	1550	120	1261-1725	1440	2540	-1279 – -815
VAL-76	gyttja/mud	Hel-1116	1.67 - 1.73	4790	130	5277-5771	5490	5340	-63 – +431
VAL-76	gyttja/mud	Hel-1117	2.06 - 2.12	7310	110	7947-8354	8150	6200	+1747 – +2154
VAL-76	gyttja/mud	Hel-1118	2.31 - 2.37	8140	120	8704-9427	9140	7100	+1604 – +2327
VAL-76	humic	Hel-1120	0.85 - 0.95	1000	100	724-1091	890	2540	-1816 – -1449
VAL-76	humic	Hel-1121	1.67 - 1.73	3100	120	2969-3571	3310	5340	-2371 – -1769
VAL-76	humic	Hel-1122	2.06 - 2.12	6820	110	7487-7867	7670	6200	+1287 – +1667
VAL-76	humic	Hel-1123	2.31 - 2.37	7440	120	8005-8446	8280	7100	+905 – +1346
VAL-77	gyttja	Hel-1441	mean at 0.37	970	120	678-1091	860	950	-272 – +141
VAL-77	gyttja	Hel-1442	mean at 0.67	1550	110	1284-1699	1450	1800	-516 – -101
VAL-77	gyttja	Hel-1443	mean at 0.95	2810	120	2736-3251	2925	2800	-64 – +451
VAL-77	gyttja	Hel-1444	mean at 1.23	3870	110	3967-4579	4290	3770	+197 – +809
VAL-77	gyttja	Hel-1445	mean at 1.55	4950	140	5446-5951	5740	4780	+666 – +1171
VAL-77	gyttja	Hel-1446	mean at 1.87	6100	110	6725-7254	7020	5770	+955 – +1484
VAL-77	gyttja	Hel-1447	mean at 2.16	7140	110	7729-8179	7950	6800	+929 – +1379
VAL-77	humus	Hel-1448	mean at 0.37	980	120	648-1095	860	920	-272 – +175
VAL-77	humus	Hel-1449	mean at 0.67	2180	100	1923-2358	2210	1800	+123 – +558
VAL-77	humus	Hel-1450	mean at 0.95	2980	100	2879-3380	3130	2800	+79 – +580
VAL-77	humus	Hel-1451	mean at 1.23	3830	110	3920-4521	4280	3770	+150 – +751
VAL-77	humus	Hel-1452	mean at 1.55	5030	140	5573-6031	5780	4780	+793 – +1251
VAL-77	humus	Hel-1453	mean at 1.87	6060	100	6672-7171	6900	5770	+902 – +1471
VAL-77	humus	Hel-1454	mean at 2.16	7140	160	7671-8223	8000	6800	+871 – +1423

Table 2. Radiocarbon dates (samples TA1-TA15 conventional radiocarbon ages and NA1-NA11 AMS-radiocarbon ages) from the sediment samples of Lake Nautajärvi. Presented are also the varve ages based on Ojala and Tiljander (2003) and the deviations between ^{14}C dates and the varve chronology.

Sample	Type	Lab ID	Depth (m)	^{14}C age BP	^{14}C age ±	^{14}C cal BP (95.4%, 2s)	Most likely ^{14}C age (cal BP)	Varve age (cal BP)	Age deviation (yrs)
TA1	clay gyttja	Su-3604	32,25	780	50	657-791	711	220	+437 – +571
TA2	clay gyttja	Su-3605	44,5	1160	50	961-1183	1083	390	+571 – +793
TA3	clay gyttja	Su-3606	61,75	1480	50	1296-1421	1372	710	+586 – +711
TA4	clay gyttja	Su-3607	80,4	1870	40	1712-1890	1809	1075	+637 – +815
TA5	clay gyttja	Su-3608	111,1	2220	50	2124-2342	2230	1575	+549 – +767
TA6	clay gyttja	Su-3609	139,25	2770	50	2762-2978	2868	2080	+682 – +898
TA7	clay gyttja	Su-3610	175,4	3220	40	3368-3513	3439	2710	+658 – +803
TA8	clay gyttja	Su-3611	214	3950	40	4285-4453	4411	3460	+825 – +993
TA9	clay gyttja	Su-3612	263,5	4460	50	4958-5296	5119	4355	+603 – +941
TA10	clay gyttja	Su-3613	314,75	5190	40	5891-6017	5948	5230	+661 – +787
TA11	clay gyttja	Su-3614	382,75	6210	40	7004-7245	7102	6380	+624 – +865
TA12	clay gyttja	Su-3615	437,5	7270	50	7995-8179	8091	7420	+575 – +759
TA13	clay gyttja	Su-3616	450,75	7630	50	8370-8540	8427	7650	+720 – +890
TA14	clay gyttja	Su-3617	501,5	8450	50	9401-9538	9477	8490	+911 – +1048
TA15	clay gyttja	Su-3618	555,1	9590	90	10686-11197	10936	9310	+1376 – +1887
NA1	clay gyttja	LuS8571	463	7875	60	8548-8811	8699	7955	+593 – +856
NA2	clay gyttja	LuS8572	466	8110	65	8931-9270	9057	8005	+926 – +1268
NA3	clay gyttja	LuS8573	469	8230	55	9027-9323	9200	8055	+972 – +1268
NA4	clay gyttja	LuS8574	472	8065	65	8699-9136	8965	8105	+594 – +1031
NA5	clay gyttja	LuS8575	475	8405	65	9277-9531	9428	8155	+1122 – 1376
NA6	clay gyttja	LuS8576	478	8300	55	9129-9453	9313	8205	+924 – +1248
NA7	clay gyttja	LuS8577	481	8590	60	9480-9688	9557	8255	+1225 – +1433
NA8	clay gyttja	LuS8578	484	8145	60	8987-9289	9097	8305	+682 – +984
NA9	clay gyttja	LuS8579	487	8455	60	9397-9542	9476	8355	+1042 – +1187
NA10	clay gyttja	LuS8580	490	8375	60	9252-9524	9395	8405	+847 – +1119
NA 11	clay gyttja	LuS8581	493	8710	60	9545-9890	9677	8455	+1090 – +1435

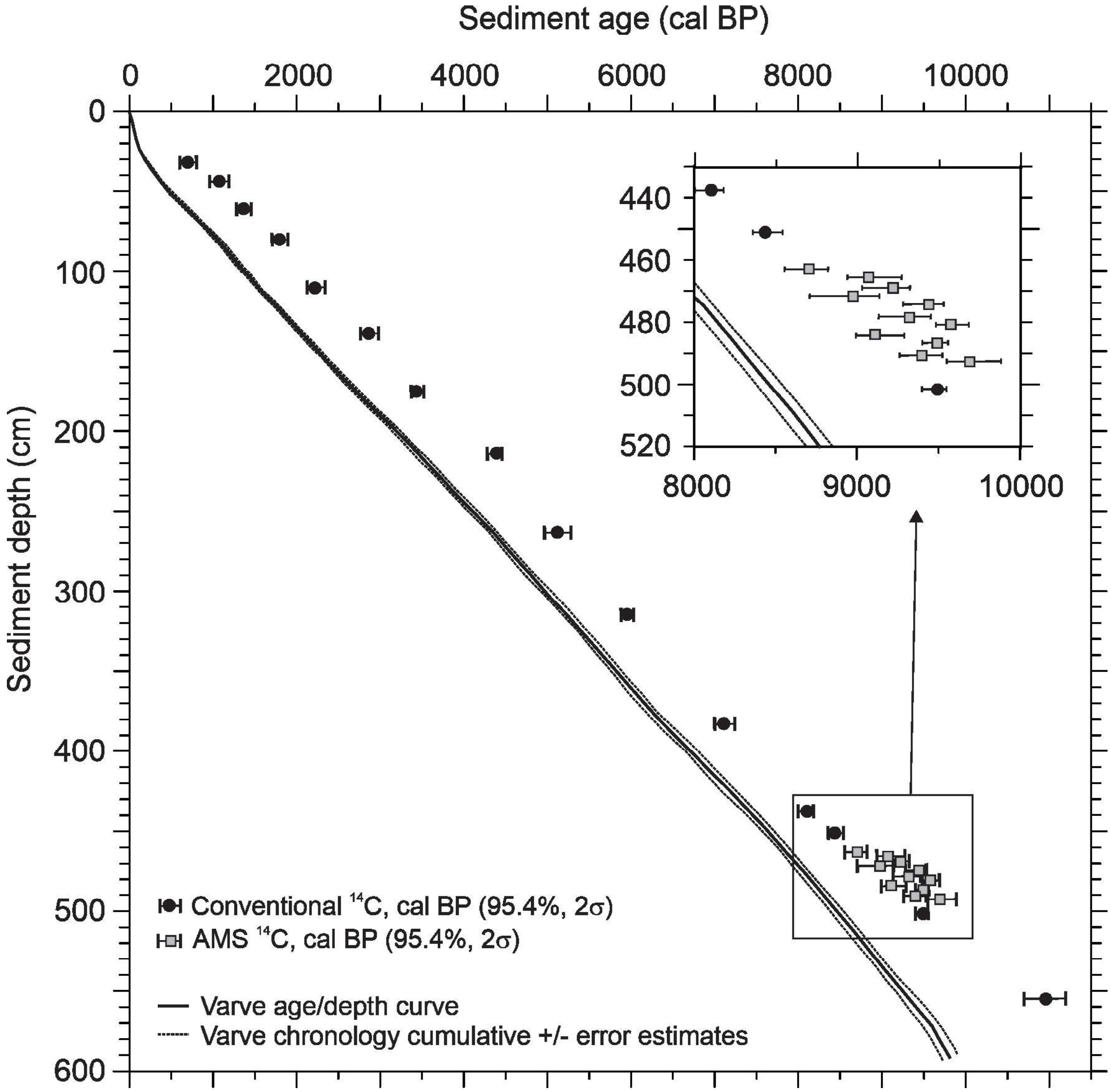

Figure 3. Varve counting-based age-depth model for Lake Nautajärvi sequence with ±1% error estimates (see Ojala and Tiljander, 2003) compared with calibrated radiocarbon mean ages for bulk conventional and AMS samples.

or AMS) (Fig. 3). On average, the calibrated radiocarbon dates are ca. 900 years older than their varve equivalents and most deviation occurs in older sediments. An attempt to wiggle match the AMS ages (e.g. Mellström et al., 2013) did not work since the reservoir age was not constant enough to identify established wiggles in the ^{14}C calibration curve.

4. Discussion

4.1. Reservoir effect in hypoxic lakes

Comparisons between varve counts and ^{14}C dates in varved lake sediments found in central Sweden show similar inconsistent deviations as the results for lakes Nautajärvi and Valkiajärvi. These lakes have also been permanently or seasonally stratified with anoxia in near bottom water for most of their history (>9000 years), and the AMS ^{14}C analyses were made from terrestrial plant macrofossils sieved from bulk sediment samples in the study of Furskogstjärnet and Mötterudstjärnet (Zillen et al., 2003; Mellström et al., 2015) and from bulk gyttja samples in Lake Kälksjön (Stanton et al., 2010). In Furskogstjärnet, the deviations between ^{14}C ages and varve chronology vary from -1118 to +495 years and in Mötterudstjärnet both dating tools give nearly similar results for sediment younger than about 6000 years, whereas in the older sequence all ^{14}C dates are older with a maximum offset of about 750 years. In the Kalksjön case, all calibrated ^{14}C ages are about 200 to 1100 years older than varve counts, thus showing the influence of the bulk sediment reservoir effect. It is noteworthy that the above-mentioned Swedish lakes have accumulated clastic-biogenic type of lacustrine varves, which is typical for several of the lakes in Finland as well (Tiljander et al., 2003; Ojala & Alenius, 2005). Reported chronological uncertainties associated with counting of sedimentary varves with this type of sequences generally fall in the magnitude of ±1–2%, which is exceptionally good with sediments (Ojala et al., 2012). In addition, the chronology of these sequences has been verified with measurements of paleosecular and paleo-intensity variations of the Earth's magnetic field (Snowball et al., 2010; Mellström et al., 2015).

The ^{14}C reservoir effect of bulk sediments in Swedish lakes was estimated to 300-400 years by Olsson (1986), although no direct studies with millennial-long varve chronologies were available then. As shown above, the reservoir effect in permanently or/and seasonally anoxic lakes can be much greater, but the varying radiocarbon results from the sediments need additional explanations, which may be related to carbon cycling in the sediment and especially to the carbon cycling in the sediment water. Perhaps some explanation for deviating radiocarbon dates can be found in carbon cycling in the surface waters of sedge mires, although the carbon cycling in mire waters is influenced by oxidation of plant remains (Billett et al., 2013; Väliranta et al., 2014). The chemical processes in the sediments of Lake Valkiajärvi that are related to carbon, however, take place in conditions where oxygen is totally lacking from the monimolimnion.

A further reasoning of the deviating ^{14}C dating results originates from the dating of the bottom near water of Lake Valkiajärvi. Soluble fraction SOL of the water yielded an age of 560 ±80 BP (Hel-1917) and the organic matter INS obtained by filtering the same water sample was 2070 ±140 (Hel-1973) ^{14}C years old (Table 1). These results perhaps explain differences observed in ^{14}C dates of the sediment and may reflect slow exchange of carbon between the atmosphere and the sediment through the monimolimnion. The above questions need, however, further investigation.

The too old radiocarbon dates in comparison to varve counts were often explained by reworked old organic material from the catchment of the lake, as in the early studies of the meromictic lakes in Lammi (e.g. Saarnisto et al., 1977). For example, in the Lake Lovojärvi case, the discrepancy was between 720 and 980 years between varve counts and conventional ^{14}C dates from bulk sediment samples (Saarnisto et al., 1977; Huttunen & Tolonen, 1977). Reworked organic material may be a valid explanation for too old radiocarbon dates at sites where anthropogenic impact, such as clearance of forest in the catchment, can be seen as primary reason for increased rate of sedimentation and impact of charcoal and in the pollen stratigraphy of the sediment. However, the too old ^{14}C dates are characteristic also for pre-land use times when direct human influence is excluded and thus other explanations are necessary. A common consequence is, however, that the lake is meromictic or at least seasonally anoxic.

Importantly, we note that in all published cases where radiocarbon dates have been compared with varve chronology or otherwise produced and secured sediment age-depth models in Finland and Sweden, the chronological deviations due to the reservoir effect and anthropogenic land-use are varied and should be clearly handled case-specific. This variation means that, in practice, it is not possible to use any fixed and/or generalized correction factor with bulk radiocarbon dates of sediments in lakes with seasonal or permanent anoxia in near bottom water.

With today's access to AMS dating one should use identified macrofossils whenever available. Nevertheless, combined with wiggle matching (Mellström et al., 2013) reliable results have been obtained in a case with rather small and constant reservoir ages. Conventional dates on bulk samples may be more suitable for studying erosion and recycling of organic material. The loss of ignition (LOI) values may reflect the influence of erosion and recycling. But in the case of Lake Nautajärvi the LOI values do not correlate with the age differences (Ojala and Tiljander, 2003).

Based on present results from lakes Nautajärvi and Valkiajärvi, as well as several examples from Sweden, it is clear that there is no constant pattern of bulk radiocarbon dates being offset with sediment depth or age, which would allow us to apply a well-defined correction factor in ^{14}C age-depth modelling. We consider that miss-interpretations may arise if problems related to radiocarbon dates from sediments of permanently or seasonally hypoxic lakes are not fully understood and acknowledged. Therefore, we consider that, for example, recent interpretations on the beginning of agriculture history of the Lammi area, southern Finland and elsewhere in Finland (e.g. Lahtinen et al., 2016 and references therein) should be considered with caution since site-specific reservoir effects age estimates derived from bulk sediment ^{14}C dates are not sufficiently resolved.

4.2. General remarks on the dating of the history of land use

Although correction factors have recently been used in statistical treatments of selected bulk radiocarbon dates to estimate, for example, the start of agriculture in Finland (Lahtinen & Rowley-Conwy, 2013; Lahtinen et al., 2016), it should be stressed that radiocarbon dates from sediments of holomictic lakes or Sphagnum (which are commonly used dating materials) are probably valid. For example, radiocarbon dates from holomictic Lake Työtjärvi in Lahti were compared with dates from an ombrotrophic raised bog Varrassuo on the shore of the lake (Donner et al., 1978). The dates from changes of pollen stratigraphy are nearly of the same age, thus suggesting that bulk sediments from holomictic lakes are as suitable as ombrotrophic *Sphagnum* peat for reliable dating (Donner et al., 1978). Blaauw et al. (2004) have shown that bulk peat samples from raised bogs (i.e. *Sphagnum* peat) do not need any corrections for the reservoir effect. A similar study has been performed in the Abisko area, northern Sweden using AMS-radiocarbon dating technique (Barnekov et al., 1998). *Sphagnum* appears as reliable material for dating also by using AMS-technique (Jungner et al., 1995; Nilsson et al., 2001). AMS ^{14}C dates from fen peat sequences, however, have resulted in surprisingly inconsistent results which are not yet fully understood as shown by Väliranta et al. (2014) in a study of fen peat in Finnish Lapland including a pure Sphagnum moss sample. Recent studies by Billet et al. (2013) on carbon cycling in peat and surface waters of mires have resulted in deviating radiocarbon ages for CO_2 and CH_4, as discussed by Väliranta et al. (2014).

All the examples presented above show that the accuracy of chronology is essential in paleo-ecological studies of human activity, which are recorded in pollen stratigraphy of lake and peat sediments. As suggested by Zolitschka et al. (2015), the most reliable chronology is achieved with the use of several independent dating methods that are applied to cross-check the published chronology and to estimate errors associated with the age-depth model (e.g. Stanton et al., 2010). This applies even for the best quality varve chronologies. In Finland, some studies have enhanced their chronological framework via using several independent dating methods within a single site. In the Orijärvi case (central Finland), for example, the pollen stratigraphy from the lake sequence was dated by a combination of AMS-radiocarbon analysis of barley and cereal grains from the nearby ancient agricultural fields and paleosecular variation of the

Earth's magnetic field recorded in the sediment (Alenius et al., 2008). In the Vuonninen case, northern Russian Karelia, the paleomagnetic dating results were considered to be more reliable than the two AMS radiocarbon dates from bulk samples with low organic content (LOI 3 to 4%), which in relation to paleomagnetic dating were found to be 700–800 years too old (Alenius et al., 2012). This demonstrates the problems related to dating bulk sediment from large lakes where primary organic matter production is low, and where the sediment contains old allochthonous carbon (Olsson, 1991). Alenius et al. (2017), show another good example, where sediment age-depth model of the Lake Huhdasjärvi sediment sequences is based on a combination of AMS radiocarbon analysis, paleomagnetic dating, and analysis of anthropogenic ^{137}Cs-activity of the recently deposited sediments.

Radiocarbon analyses are made from various types of materials and therefore the reliability of the dates can vary a great deal. On top of the problematic radiocarbon dates from sediments of the meromictic soft water lakes, the ageing "hard water" effect limits the use of calcareous sediments for radiocarbon dating (Olsson, 1986). Radiocarbon dates from bulk sediment samples with low organic content from large lakes are too old because the sediments contain old allochthonous carbon. The age deviation of charcoal samples from archaeological dwelling sites can be large, in cases up to half of the dates are erroneous, in extreme cases charcoal from Stone Age fireplaces may be recent (Jungner, 1977). When paleo-ecological interpretations are made from extensive data, they should not be handled as one entity from the point of dated material. Statistical treatment will not improve the quality of the interpretations, which depends fully on the knowledge of possible reservoir offsets of single ^{14}C dates. Dating results from sediments of small holomictic lakes are often logic and comparable to dates from ombrotrophic *Sphagnum* peat which, in principle, is the most suitable material for radiocarbon analysis. In the study of land use history bulk sediment samples are still often used which creates a risk of reworked old organic matter from the lake's surrounding as observed in the Lammi area (Saarnisto et al., 1977; Huttunen & Tolonen, 1977). AMS-radiocarbon dates from single terrestrial macrofossils are well known to be ideal for reliable chronology (Vanhanen et al., 2019). Therefore, it is strongly recommended to critically consider the quality and outcome from different age models even in published papers in order to avoid biased conclusions about land use.

5. Conclusions

The above results from Nautajärvi and Valkiajärvi show that bulk sediments of meromictic or seasonally anoxic lakes are not suitable for bulk radiocarbon dating, because the dating results deviate inconsistently from calendar years, i.e. the reservoir effect is not constant. This conclusion became evident already late in the 1970´s in the study of several meromictic lakes in the Lammi area, southern Finland, and was confirmed based on studies in central Sweden (Zillen et al., 2003; Stanton et al., 2010). The radiocarbon dates from the bulk sediments of hypoxic lakes are inconsistent when compared to varve years and inconsistent between soluble and insoluble fractions. The dating trends can also vary greatly over time in the same lake. The radiocarbon reservoir effect in the hypoxic lakes with seasonal or non-existent overturns probably explains most of the deviating bulk radiocarbon dates. In addition, the role of carbon cycling in the sediment may play an important role for dates, as seems to be the case in surface waters of peatlands. The radiocarbon dates from the bulk sediments of meromictic lakes should not be used for dating paleo-ecological events in the history of the lake basin and its surroundings including the history of land use, especially the commence of agriculture.

Thus, the bulk radiocarbon dates from sediments of meromictic lakes should not be utilized for dating as long as offsets cannot be quantitatively explained. It is important that geologists, physical geographers but also archaeologists and paleo-ecologists are aware of these challenges and consider the significant uncertainties in their interpretations. Advanced statistical treatment of radiocarbon dates will not lead to improved chronology as long as the nature of the dated material behind the numbers is not understood.

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