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Total organic carbon (TOC) of lake water during the Holocene inferred from lake sediments and near-infrared spectroscopy (NIRS) in eight lakes from northern Sweden

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Abstract. The aim of this study is to infer past changes in total organic carbon (TOC) content of lake water during the Holocene in eight boreal forest, tree-limit and alpine lakes using a new technique – near-infrared spectroscopy (NIRS). A training set of 100 lakes from northern Sweden covering a TOC gradient from 0.7 to 14.9 mg l^{-1} was used to establish a relationship between the NIRS signal from surface sediments (0–1 cm) and the TOC content of the water mass. The NIRS model for TOC has a root mean squared error (RMSECV) of calibration of 1.6 mg l^{-1} (11% of the gradient) assessed by internal cross-validation (CV), which yields an R^2_{cv} of 0.61. The results show that the most dramatic change among the studied lakes occurs in both tree-line lakes around 1000 yrs BP when the TOC content decreases from ca. 7 to 3 mg l^{-1} at the present, which is probably due to a descending tree-limit. The TOC content in the alpine lakes shows a declining trend throughout most of the Holocene indicating that TOC may be more directly correlated to climate in alpine lakes than forest lakes. All boreal forest lakes show a declining trend in TOC during the past 3000 yrs with the largest amplitude of change occurring in the lake with a connected mire. The results indicate that a change to a warmer and more humid climate can increase the TOC levels in lakes, which in turn may increase the saturation of CO_2 in lake waters and the emission of CO_2 to the atmosphere.

Introduction

One of the most important challenges remaining to be addressed by climatologists is the mechanism responsible for the observed coherence between climate change and carbon cycling. Several studies have shown that most lakes in the world are net sources of carbon dioxide (CO_2) to the atmosphere (Kling et al. 1991; Cole et al. 1994; Algesten et al. 2004) and that the CO_2 emission from lakes is proportional to the input and lake mineralization of terrestrial organic carbon (Kling et al. 1991, 1992; Hope et al. 1996; Sobek et al. 2003). Between 30 and 80% of the total organic carbon that enters freshwater ecosystems is lost in lakes due to mineralization, and subsequent CO_2 emission to the atmosphere is by far the most important carbon loss process (Algesten et al. 2004). There are numerous projects that have aimed to increase our understanding of present-day carbon and nutrient cycling in boreal and arctic ecosystems; however, this research is usually limited to studies of small-scale, shorter-term variation at one single site.

Long-term variations in the past environmental conditions of lakes and their catchments can be inferred using the physical, chemical and biological records preserved in lake sediments. The most common tools for reconstructing long-term changes in past carbon and nitrogen cycling in lake systems are analyses of carbon and nitrogen (C, N, $\delta^{13}C$, $\delta^{15}N$ and C/N ratios) and diatoms (MacDonald et al. 1993; Hammarlund et al. 1997; Korsman and Segerström 1998; Wolfe et al. 1999, 2003). Chemical analyses can be difficult to interpret because catchment hydrology, erosional inputs of terrestrial organic matter, and gas exchange are all important factors. While diatoms are known to be sensitive indicators of pH (Renberg et al. 1993; Battarbee et al. 1999), models for diatom-inferred TOC (Korsman and Birks 1996; Rosén et al. 2000a) generally show rather poor statistical performance and TOC may be confounded by the pH signal. Therefore, there is a need for new, independent methods and multi-proxy approaches to facilitate interpretation of palaeorecords and to improve our understanding of the effects of climate change in relation to carbon cycling.

This study tests the hypothesis that near-infrared spectroscopy (NIRS) of lake sediments can be used to infer the total organic carbon content in lake water during the Holocene. This is logical because lake sediments constitute a mixture of proteins, lipids, carbohydrates and other biochemicals derived from tissues of organisms formerly living in a lake and its catchment (Meyers and Ishiwatari 1993). Humic substances are diagenetically formed from these starting materials and they are a major component of the organic content of lake water and sediment. NIRS utilizes the fact that the organic fraction of lake sediment has a distinctive but complex NIR signature that can be summarized using multivariate statistical tools. Results from a training set in northern Sweden showed that there is a relationship between the NIRS signal of surface sediment and the TOC content in the water mass (Nilsson et al. 1996). The training set in that study was rather small ($n = 25$ lakes) and was never tested for retrospective analysis. This study uses a training set of 100 lakes for a more rigorous test and the model is applied to eight Holocene sediment cores from boreal forest, tree-limit and alpine lakes.

Material and methods

Study area

The training set of 100 lakes from northern Sweden (67°07′–68°48′ N) has a total lake-water organic carbon (TOC) content that spans from 0.7 to 14.9 mg l^{-1}, a range in altitude from 169 to 1183 m a.s.l., annual precipitation rates from 300 to 1900 mm yr^{-1} and mean July air temperature from 7 °C to

14.7 °C (Alexandersson et al. 1991). The correlation between the TOC content of the training-set lakes and altitude is $R^2 = 0.44$, and precipitation generally increases and summer temperature decreases with altitude. The lakes are mainly small (< 20 ha) headwater lakes situated on similar bedrock (mainly granite and gneiss), and with maximum depths ranging from 1.5 to 16 m. All lakes are from an area with low human impact.

Eight lakes were used for down-core reconstruction (Table 1; Figure 1). Lake Makkasjön, Lake Lundsjön and Lake Sotaure (unofficial name) are situated in a nutrient-poor, pine heath, kettle-hole area dominated by Scots pine (*Pinus sylvestris* L.) of Ericaceae-Cladina type mixed with Norway spruce (*Picea abies* (L.) Karsten) and some isolated birch (*Betula pubescens* L.). According to Jokkmokk Commonage, Makkasjön and Lundsjön were treated with rotenone in 1965 (0.3 mg l^{-1}) and with a continuous stock of Arctic char (*Salvelinus alpinus* L.) since then. Makkasjön was also treated with superphosphate ($Ca(H_2PO_4)_2$) in 1990 and limed (3 tonnes of dolomite lime). Prior to liming Makkasjön had a pH of 5.5, alkalinity 3 μeq l^{-1}, colour 10 mg Pt l^{-1} and total phosphorus 4 μg l^{-1}. In October 1990 (1 year after limning) the pH was 6.4 and in March 1992 the pH was 5.6 and the alkalinity 120 μ eq l^{-1}.

The catchments of Lake Seukokjaure (unofficial name) and Lake Tsuolbmajavri are situated on gentle slopes at the tree line with only scattered mountain birch trees in the catchment. Lake 850, Lake Njulla and Lake Niak (unofficial names) are situated on alpine heath with sparse catchment vegetation (mainly lichens and mosses) and little or no soil. There is neither forestry nor cultivated land in the catchment areas.

Field and laboratory methods

To assess the relationship between the NIRS signal of lake sediments and lake-water TOC, surface-sediments (0–1 cm) were sampled during the summers in 1997–1998 from the deepest part of each lake using a gravity corer (Renberg 1991); HTH Teknik, Vårvägen 37, SE-951 49 Luleå. Water samples for TOC measurements were taken from 1 m water depth at the same occasion as the sediment sampling using a Limnos-type water sampler. Water chemistry was analysed by an accredited laboratory within 5 days of collection (Department of Environmental Analysis, SLU Uppsala and Miljölaboratoriet, Umeå). The Holocene sediment cores were taken from the deepest part of each lake (Table 1) using a Russian peat or Livingston corer. Surface sediment cores were taken using a gravity corer. The sample interval was 1 cm for all lakes except for Niak where every 5 cm was analysed.

Prior to NIRS analysis about 2 cm^3 of wet sediment was freeze-dried and ground in a mortar. NIR spectra were recorded using a NIRSystems 6500 instrument (FOSS NIRSystems Inc., Silver Spring, MD, USA). The instrument measures diffuse reflectance (R), which is transformed to apparent absorbance

Table 1. Location and environmental characteristics of the study lakes.

Variables	Makkasjön	Sotaure	Lundsjön	Tsuolbmajavri	Seukokjaure	L-850	Njulla	Niak
Latitude (° N)	66°43′ N	66°43′ N	66°42′ N	68°41′ N	67°46′	68°18′	68°22′	67°30′
Longitude (° E)	20°35′ E	20°36′ E	20°36′ E	22°05′ E	17°31′	19°07′	18°42′	17°31′
Altitude (m a.s.l.)	415	425	425	526	670	850	999	1172
Lake area (ha)	3	2	3	14	11	1.5	1	19
Drainage area (ha)	7	4	90	432	617	29	78	153
Drainage area/lake area	2.3	2	30	31	56	19	78	8
Catchment vegetation	boreal forest	boreal forest	boreal forest	at treeline	at treeline	alpine	alpine	alpine
Lake treatment	R. L. S. FI.	none	R. L. FI.	none	none	none	none	none
Bedrock	granite	granite	granite	Quarzite/granodiorite	granite/syenite			amphibolite/granite
Max. depth (m)	15	9	8	5	6	8	4	13.2
pH (units)	6.1	6.2	6.2	7.4	–	6.8	6.8	6.4
TOC (mg l^{-1})	3.1	4.5	3.3	4.9	–	2.4	1.6	1.9
DOC (mg l^{-1})	2.9	4.6	3.3	–	–	2.3	1.0	1.9
Sediment core length (cm)	124	70	121	281	141	104	230	210
Sampling year	1995	2003	2003	1997	1996	1999	1999	2000

R = rotenone treatment, L = limed, S = superphosphate, FI. = fish introduction.

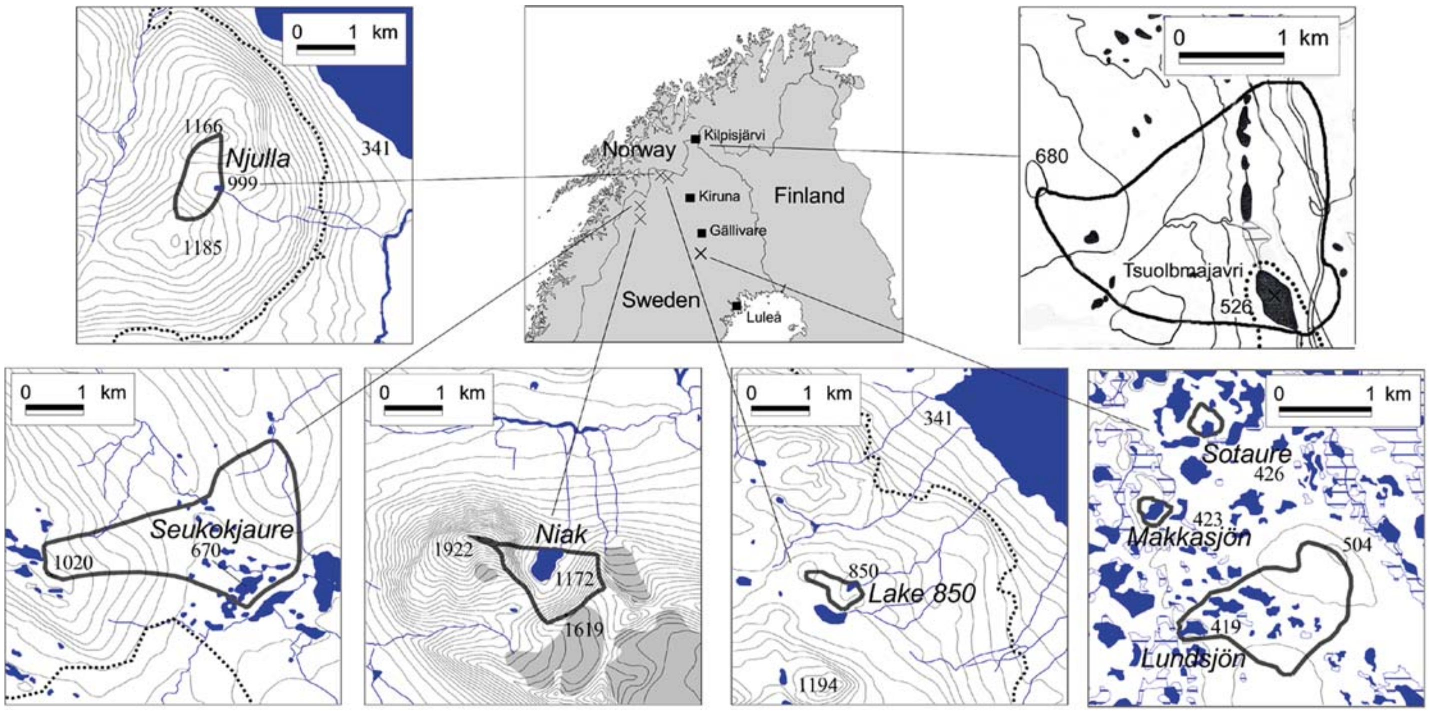

Figure 1. Map showing the study area and the eight study lakes Njulla, Seukokjaure, Niak, L-850, Lundsjön, Makkasjön, Sotaure and Tsuolbmajavri. The thick lines indicate the drainage areas, the dotted lines indicate the birch forest limit and the shaded areas indicate glaciers.

values (A) according to $A = \log(1/R)$. Data were collected at 2-nm intervals between 400 and 2500 nm yielding 1050 data points per sample.

Dating

Radiocarbon dating of terrestrial macrofossils, *Drepanocladus* moss and bulk sediment samples was performed using accelerator mass spectrometry (AMS) (Ångström Laboratory, Box 534, SE-752 21 Uppsala and Dating Laboratory of the University of Helsinki (Tsuolbmajavri)). For Makkasjön and Lundsjön approximate dates in recent sediment layers were also assessed using concentrations of spheroidal carbonaceous fly ash particles (SCP) (Renberg and Wik 1985; Wik and Renberg 1996). The SCP show that 7 cm depth in Makkasjön and 8 cm depth in Lundsjön correspond to the increase in fossil fuel combustion following the Second World War (ca. 1950 A.D.) and the maximum SCP peak at 2 cm (ca. 1200 SCP g^{-1} dry sediment for Makkasjön and 2300 SCP g^{-1} dry sediment for Lundsjön) corresponds to the peak in oil consumption in the early 1970s. No SCP were recorded below 8 cm depth in any lake. Changes in the $^{206}Pb/^{207}Pb$ ratio and lead concentration at 30 cm depth in Makkasjön correspond to a general increase in the atmospheric fall-out of pollution lead in northern Europe at ca. 1000 yrs BP Renberg et al. 2001). At 42 cm depth the *Picea* pollen reach 5% in Makkasjön, which is usually seen as the limit for when *Picea* is established in the catchment area; the establishment of *Picea* in northern Scandinavia is dated to around 3000–2500 cal. BP (Tallantire 1972, 1977; Segerström 1990). The high abundance of *Alnus* at 120 cm depth in Makkasjön corresponds to ca. 9000 cal. BP when *Alnus* dominated the landscape following deglaciation (Tallantire 1977; Huntley and Birks 1983). It is assumed that the sedimentation of organic matter in Sotaure started around the same time as in Makkasjön and Lundsjön since these lakes are very close to each other (2 km) and at a similar altitude. This is supported by the ^{14}C date at 124 cm in Lundsjön (Table 2).

Numerical analyses

For detection of outliers principal component analysis (PCA) (cf. Jackson 1991) was used on multiplicative signal corrected (MSC) (Geladi et al. 1985; Martens and Næs 1989) NIR spectra. TOC measurements were regressed on NIR spectra of surface sediment samples by partial least square regression (PLS) (e.g. Martens and Næs 1989). PLS regression summarises the numerous independent wavelengths from the NIR spectra into a few orthogonal components.

The best model was selected as the one that produced the lowest root mean squared error of calibration (RMSECV) and the best R^2_{cv} assessed by internal

Table 2. Approximate dates from Makkasjön, Lundsjön, Sotaure, Seukokjaure, Tsuolbmajavri, L-850, Njulla and Niak using spheroidal carbonaceous particles (SCP) from fossil fuel combustion [Renberg and Wik 1985], lead pollution history [Renberg et al. 2001], vegetation development [Tallantire 1977; Segerström 1990; Huntley and Birks 1983] and radiocarbon dates.

Lake	Material	Sediment depth (cm)	Dates (^{14}C yrs. BP)	Calibrated age (yrs. BP)
Makkasjön	SCP	2	–	35
Makkasjön	SCP	7	–	55
Makkasjön	$^{206}Pb/^{207}Pb$	30	–	1000
Makkasjön	Picea	42	–	2500
Makkasjön	Alnus	120	–	9000
Lundsjön	SCP	2	–	35
Lundsjön	SCP	8	–	55
Lundsjön	TM	63	3840 ± 45	4410–4090
Sotaure	TM	124	8085 ± 60	9210–8715
Sotaure	TM	26	4100 ± 40	4820–4440
Seukokjaure	TM	111	7260 ± 75	8160–7910
Tsuolbmajavri	Bulk	20–21	1369 ± 55	1190–1310
Tsuolbmajavri	Bulk	40–41	2090 ± 55	1950–2120
Tsuolbmajavri	DM	60–61	2435 ± 55	2350–2710
Tsuolbmajavri	DM	80–81	2520 ± 70	2360–2740
Tsuolbmajavri	Bulk	100–101	3070 ± 55	3080–3370
Tsuolbmajavri	DM	120–121	3670 ± 75	3840–4140
Tsuolbmajavri	DM	140–141	4115 ± 75	4440–4820
Tsuolbmajavri	Bulk	160–161	4515 ± 85	4990–5310
Tsuolbmajavri	Bulk	180–181	4900 ± 85	5590–5710
Tsuolbmajavri	Bulk	200–201	5355 ± 85	5990–6280
Tsuolbmajavri	Bulk	230–231	6680 ± 90	7440-7550
Tsuolbmajavri	Bulk	250–251	8060 ± 95	8720–9140
Tsuolbmajavri	Bulk	270–271	8280 ± 80	9050–9420
Tsuolbmajavri	Bulk	290–291	10940 ± 95	12760–12960
L-850	Bulk	11	1030 ± 400	1400–600
L-850	Bulk	31	2690 ± 400	3500–2350
L-850	Bulk	56.5	4075 ± 80	4835–4440
L-850	Bulk	74.5	5000 ± 85	5885–5650
L-850	Bulk	93	6290 ± 400	7650–6750
L-850	Bulk	117.5	8525 ± 80	9550–9434
Njulla	TM	57.5	1520 ± 80	1520–1280
Njulla	TM	76.5	2295 ± 85	2750–2050
Njulla	TM	108.5	3585 ± 80	4100–3640
Njulla	TM	162.5	7520 ± 135	8600–8100
Njulla	TM	207.5	8505 ± 130	9900–9100
Niak	Bulk	210	7810 ± 170	9140–8200

Calibrated ages are within 2σ. The indirect dates are based on studies using radiocarbon dating (calibrated 14C-dates) or annually laminated sediments. TM = terrestrial macrofossil, Bulk = bulk sediment, DM = *Drepanocladus* moss.

cross-validation (CV) with 10 groups. RMSECV was calculated according to Equation (1)

$$RMSECV = \frac{1}{I}\sqrt{\frac{\sum_{i=1}^{I}(y_i - \hat{y}_i)^2}{\sum_{i=1}^{I}(y_i - \bar{y})^2}} \tag{1}$$

where I is the number of lakes, y_i is the measured TOC for lake i, y is the mean TOC for all lakes and $\hat{y}_i$ is the predicted TOC. The SIMCA-P 10.0 (Umetrics AB, SE-901 19 Umeå, Sweden) software was used for all multivariate data analysis.

Results and discussion

Modern calibration of NIRS

The results show that NIRS spectra from lake sediment contain information on the TOC content of the water mass. A 4-component PLS model gives a RMSECV of 1.6 mg l^{-1} (11% of gradient) and an R^2_{cv} of 0.61 between NIR spectra of the surface sediment and the TOC content in lake water in the 100-lake calibration set. Due to the remoteness of the lakes the calibration is based on single TOC measurements for most lakes sampled during 1997–1998, which includes values from late July to September. For 15 of the lakes, TOC was measured 8–10 times during 1998–1999 and the variation for most lakes was in the range of the RMSECV for the model of 1.6 mg/l (range 0.5–3.8 mg/l) (Karlsson 2001). The TOC values from our measurements are therefore not fully comparable between lakes. For retrospective analysis, it is important to include the full range of conditions that have occurred during the period of interest (Birks 1998) and, therefore, all 100 lakes were included in the calibration. Future improvements should include a better estimate of the TOC content in the calibration lakes.

Reconstruction of past total organic carbon (TOC) of lake water using NIRS

Correlation between NIRS and LOI and potential degradation effects

It can be argued that the NIRS-inferred TOC only reflects the organic content of the sediment (as inferred from loss-on-ignition) because, NIRS can be sensitive to the organic content as well as the organic composition of sediment like proteins, lipids, carbohydrates and other biochemicals derived from tissues of organisms formerly living in a lake and its catchment. However, a correlation coefficient (R^2) from 0.00–0.78 between NIRS-inferred TOC and LOI in the eight Holocene sediment cores indicates that NIRS can capture a large variation that cannot be accounted for solely by LOI. It is well established that the NIRS signal is sensitive to the composition of organic material and not only content (Bokobza 1998), which is supported by a whole-basin study based on surface sediments (Korsman et al. 1999).

There is no indication that the NIRS signal in the uppermost part of the sediment is affected by degradation processes. The NIRS-inferred TOC from the surface sample is for Makkasjön 3.5 mg l^{-1} (measured 3.1 mg l^{-1}), Sotaure 4.1 mg l^{-1} (4.5 mg l^{-1}), Lundsjön 6.0 mg l^{-1}(3.3 mg l^{-1}), L-850 3.2 mg l^{-1} (2.4 mg l^{-1}) and Njulla 1.3 mg l^{-1} (1.6 mg l^{-1}). Considering that the TOC varies in the range 0.5–3.8 mg/l during the season in the region (Karlsson 2001) the differences between inferred and measured TOC is small for all lakes (0.3–0.3 mg l^{-1}) except for Lundsjön (0.8 mg l^{-1}). The other lakes have no present day value. The reconstructed lakes were not included in the calibration set. Makkasjön, Lundsjön and Seukokjaure show an increase of NIRS-inferred TOC toward the surface. In Makkasjön and Lundsjön this increase from 2.3 to 3.5 and 5.3–6 mg l^{-1}, respectively, at 3–3.5 cm depth corresponds to ca. 1965 when the lakes were treated by rotenone and stocking of fish began. Makkasjön was also treated with superphosphate and limed in 1990. The nearby lake Sotaure is untreated and does not show an increase in inferred TOC towards the surface. The increase in NIRS-inferred TOC in Seukokjaure occurs immediately after a minerogenic layer at the top of the core. The minerogenic layer is probably a result of the construction of a gravel road next to the lake. The result is supported by a comparison of the NIRS signal of sediment from 0–1 cm and 1–2 cm in 56 lakes from the same area, where a degradation affect could not be demonstrated using partial least squares discriminant analysis (PLS-DA) (Rosén et al. 2000b). Overall, the results show that predictive transfer functions can be established between NIRS spectra and the TOC content in lake-water and be used for retrospective analysis.

NIRS-inferred TOC in boreal forest lakes

NIRS infer a gradual decrease in TOC during the last 3000 years in all three boreal forest lakes (Figure 2). There was only a small variation in the TOC content before 3000 BP in both Lundsjön and Sotaure with the exception of the initial phase after the deglaciation when NIRS infer an increase in TOC. Makksjön shows a larger variation in the TOC content and higher TOC values during the periods ca. 9000–7500 BP and ca. 4000–2500 BP. Makkasjön has the greatest extent of mire connected to the lake and the area around Makkasjön is flat; thus, a likely explanation for changes in NIRS-inferred TOC is changes in allochthonous input of carbon to the lake due to changing hydrology in the drainage area. The rapid increase in TOC around 4000 BP in Makkasjön occurs at the same level where the first *Picea* pollen was found (Korsman and Segerström 1998). It is argued that *Picea* expanded in northern

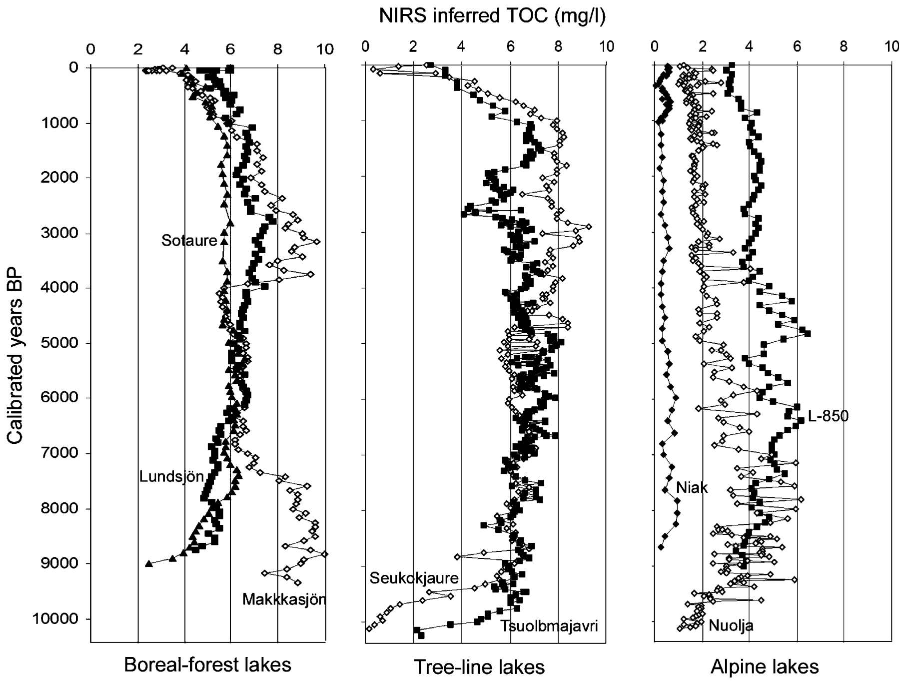

Figure 2. NIRS-inferred total organic carbon (TOC) content in lake water in Holocene sediment cores from Lundsjön, Makkasjön, Sotaure, Seukokjaure, Tsuolbmajavri, L-850, Niak and Nuolja.

Scandinavia around 3000–2500 BP when there was a climatic shift to a more humid climate (Segerström 1990), which would favour mire expansion and a greater allochthonous input of carbon to the lake. In contrast, Lundsjön has only a small mire and Sotaure no mire connected to the lake and these lakes show only small changes in TOC during the same time.

NIRS-inferred TOC in tree-line lakes

The two tree-line lakes, Seukokjaure and Tsuolbmajavri, show very similar patterns in NIRS-inferred TOC throughout the Holocene even though the lakes are about 300 km apart. A notable change occurs around 1000 BP when the NIRS-inferred TOC decreases rapidly from ca. 7 to the present-day value of 3 mg l^{-1}. About 1000 BP the forest limit reached ca. 100 m higher than today (Karlén 1976; Hafsten 1981) and during the 'Little Ice Age', from 650 to 150 BP, climate became cooler (Briffa et al. 1992) and the forest limit descended (Aas and Faarlund 2001). Due to the uncertainties with ^{14}C-dating it is not possible to set an exact date for the decline in TOC; however, the most likely interpretation is that the decline occurs during the 'Little Ice Age' and that the lakes changed from being birch-forest lakes to lakes situated just at the tree line. Today, Tsuolbmajavri has only a thin zone of birch around the lake and Seukokjaure only scattered trees in the catchment. Rosén et al. (2000b) showed that alpine and birch-forest lakes can be separated using NIRS of lake sediments and SIMCA classification based only on the quality of the organic carbon in the sediment. The clear distinction is probably due to both the amount and quality of the allochthonous carbon, which is different in forested and alpine catchments. This interpretation is also supported by Karlsson (2001), who showed that alpine lakes usually contain less TOC than lakes in the birch forest in northern Sweden.

NIRS-inferred TOC in alpine lakes

The alpine lake situated at the lowest elevation (Niak, 850 m a.s.l.) has the highest inferred TOC values and the lake situated at the highest elevation (Lake Naik, 1250 m a.s.l.) has the lowest inferred TOC values. All three alpine lakes show a slightly decreasing trend in TOC through most of the Holocene (except the initial deglaciation phase). Several studies indicate a generally decreasing temperature in Sweden during the Holocene (Kullman 1995, 1999; Kullman and Källgren 2000; Bigler et al. 2003). A warmer climate, higher insolation and higher lake-water pH in the early Holocene would have favoured higher organic production in the early Holocene, and the results here indicate that the TOC content in alpine lakes is more directly influenced by climate than forest lakes, where changes in vegetation and formation of mires also are important.

Conclusions

This study shows that the NIRS spectra of lake sediment contain information about the TOC content of lake water and that NIRS can capture long-term processes specific to different types of lakes. All eight lakes from northern Sweden show small changes or a decreasing trend in the TOC content during the last 3000 years. The results indicate that the most rapid changes in TOC occur when a lake's catchment changes from a birch forest to an alpine type environment due to a descending tree-line or when the allochthonous input of carbon to the lake changes due to changes in the extent of adjoining mires. The overall trends in TOC correspond well with a general cooling during the past 3000 years (Briffa et al. 1992; Kullman 1995; Bigler et al. 2002). Given the predicted climate change scenario with a rising tree-line and an expanding mires due to a more humid climate, the paleolimnological data indicate that TOC levels can be expected to increase in boreal and alpine lakes. Because super-saturation and emission of CO_2 from lakes is correlated to the organic carbon content in lake water (Jonsson et al. 2003; Algesten et al. 2004), higher TOC level could lead to an increased emission of CO_2 from the lakes. The data presented here are not intended to be conclusive. Considerably more sites need to be evaluated to allow for a thorough testing of the technique. However, because NIRS is a cost effective technique where about 70 samples can be analysed per day, future research can include a larger number of lakes to further assess the uniformity of the TOC reconstructions among different types of lakes. This would improve the understanding of the importance of long-term processes on the carbon cycling in lakes. Greater application of NIRS to long-term sediment records may also improve our understanding of the importance of lakes for CO_2 emissions to the atmosphere and allow us to predict future directions of change in the landscape.

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