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How well can near infrared reflectance spectroscopy (NIRS) measure sediment organic matter in multiple lakes?

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Abstract Loss-on-ignition (LOI) is the most widely used measure of organic matter in lake sediments, a variable related to both climate and land-use change. The main drawback for conventional measurement methods is the processing time and hence high labor costs associated with high-resolution analyses. On the other hand, broad-based near infrared reflectance spectroscopy (NIRS) is a time and cost efficient method to measure organic carbon and organic matter content in lacustrine sediments once predictive methods are developed. NIRS-based predictive models are most robust when applied to sediments with properties that are already included in the calibration dataset. To test the potential for a broad applicability of NIRS models in samples foreign to the calibration model using linear corrections, sediment cores from six lakes (537 samples, LOI range 1.03–85%) were used as reference samples to develop a predictive model. The applicability of the model was assessed by sequentially removing one lake from the reference dataset, developing a new model and then validating it against the removed lake. Results indicated that NIRS has a high predictive power (RMSEP < 4.79) for LOI with the need for intercept and slope correction for

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new cores measured by NIRS. For studies involving many samples, NIRS is a cost and time-efficient method to estimate LOI on a range of lake sediments with only linear bias adjustments for different records.

Keywords Holocene · Lacustrine · Paleolimnology partial least squares (PLS) · Sediment organic matter

Introduction

Near infrared reflectance spectroscopy (NIRS) is increasingly included in soil studies to reduce costs and processing time (Chodak 2008). NIRS is a non-destructive method where a light beam with known spectral properties is directed at a sample and the reflected light is measured in the visible and near infrared region of the light spectrum (approx. 350 nm to 2500 nm) (Kaye 1954, 1955). Measured reflectance values are related to the organic bonds between molecules in a sample, making it feasible to calibrate multivariate predictive models to measure organic compounds in sediment analyses. The main strength of NIRS compared to traditional physical and chemical analyses of sediments is that a single sample could be used to simultaneously analyze a range of different chemical or structural parameters (Cozzolino and Morón 2003) and since it is a non-destructive method the sample can then be used for other analyses.

The content of organic matter in sediments is widely used as a stratigraphic property and as, for example, an indicator of ecosystem productivity in lake sediment analyses as well as for assessing climate and environmental changes (Björck et al. 1991; Nesje and Dahl 2001; Birks and Birks 2006). The principal analytical method, loss on ignition (LOI), which has been used for several decades, is an accurate method to measure soil and sediment organic matter (LOI at 550 °C) and CaCO$_3$ content (LOI at 950 °C) (Dean 1974; Heiri et al. 2001) and although inexpensive in materials, it is expensive in staff time. Alternative methods using wet oxidation (Walkley–Black method; Heanes 1984) are more expensive and time consuming even when automated, and may over-estimate the actual organic carbon content (Wang et al. 2012).

NIRS has been successfully applied in previous studies to measure the physical and chemical composition of lake sediments, including LOI and geochemical elements such as C, N and P (Malley et al. 1999, 2000; Inagaki et al. 2012). The ability of this method to measure different proxies from a single analysis results in reduced sampling and laboratory analysis costs, together with increase time-efficiency (Nduwamungu et al. 2009). However, the use of NIRS has been limited to the cores (or lakes) for which the calibration models have been developed (Rosén et al. 2010). The restriction of the applicability of NIRS models to the cores, or lakes against which the models have been calibrated is a strong drawback for a wider application of the method. It has been assumed that site-specific variation in sediment chemical composition or stratigraphy will negatively affect the model applicability, even rendering it unusable in some situations. However, there is a growing literature on the applicability of spectrometric tools to samples that are outside the calibrated population (Rosén et al. 2011; Meyer-Jacob et al. 2017), although new measurements will generally require some correction factors based on a subset of samples from the new core (Roggo et al. 2007). There has been a marked development of spectroscopic tools to measure sediment properties with very good results, although transferability of models is still a remaining challenge (Table 1).

Here we present LOI predictive models based on NIRS scans of six contrasting lacustrine sediment cores from boreal to high Arctic locations to study the (1) method's accuracy and feasibility, and (2) test if the transferability of NIRS models to new samples with only a simple linear correction as adjustment.

Materials and methods

Lake sediments

A total of 537 lacustrine sediment samples were analysed from six geographically distinct lakes across the boreal to high arctic zone (Table 2). For three of the lakes, the description of the cores has already been published: Skartjørna in Svalbard (Alsos et al. 2016), Uhca Rohči at Varanger Peninsula in Northern Norway (Clarke et al. 2019), and Bolshoye Schuchye in the Polar Urals (Svendsen et al. 2018). In two cases, we sampled new cores from lakes for which earlier lithological studies exist- Gauptjern in Troms (Jensen and Vorren 2008) and Øvre Æråsvatnet in Nordland

Table 1 Examples of previous work done using spectroscopy in sediment analyses from marine and lake cores

Authors	Developed NIRS models	Method	Details
Balsam and Deaton (1996)	TOC and Carbonates	NIRS	Marine sediments, atlantic ocean. High applicability ($R^2 > 0.9$)
Malley et al. (1996)	TOC, TN, TP	NIRS	Lake sediments, 10 sites, Canada. High applicability ($R^2 > 0.88$)
McTiernan et al. (1998)	Moisture, organic matter	NIRS	Lake sediments, 1 site, England. High applicability ($R^2 > 0.9$)
Korsman et al. (1999)	LOI	NIRS	Lake sediments, 1 site, Sweden. High variability in PCA of sediment spectra explained by LOI
Malley et al. (1999)	TOC, TN, TP, diatoms and carbonates	NIRS	Lake sediments, 1 site, Germany. High applicability ($R^2 > 0.83$; TC $R^2 = 0.93$)
Mecozzi et al. (2001)	Carbonates	FTIRS	Marine sediments, Italy. High applicability ($R^2 = 0.99$)
Rosén and Persson (2006)	TOC	FTIRS	Lake sediments, 2 sites, Sweden. Limited applicability ($R^2 > 0.6$)
Leach et al. (2008)	TOC	FTIRS	Marine sediments, atlantic ocean. Good applicability ($R^2 = 0.83$)
Vogel et al. (2008)	TOC, TIC, TN, TS	FTIRS	Lake sediments, 2 sites, Russia and Albania. $R^2 > 0.79$ (TOC), $R^2 = 0.98$ (TIC), $R^2 > 0.62$ (TN), $R^2 = 0.21$ (TS)
Rosén et al. (2010)	LOI, TIC, BSi	FTIRS	Lake sediments, multiple sites, worldwide. Limited geografical transferability of models
Rosén et al. (2011)	TOC, TIC, BSi	FTIRS	Lake sediments, multiple sites, worldwide. High transferability of models ($R^2 > 0.9$)
Inagaki et al. (2012)	TOC, TN, TS, inorganic elements	FTIRS	Lake sediments, Japan, 1 site. High applicability (TOC $R^2 = 0.88$, TN $R^2 = 0.89$, inorganic elements $R^2 = 0.73$)
Hahn et al. (2013)	TOC, TIC, BSi	FTIRS	Lake sediments, Patagonia, 1 site. High applicability ($R^2 = 0.94$)
Meyer-Jacob et al. (2014)	TOC, TIC, BSi	FTIRS	Lake sediments, Russia, 1 site. High applicability ($R^2 > 0.86$)
Pearson et al. (2014)	TOC	FTIRS	Lake sediments, Japan, 1 site. High applicability ($R^2 = 0.95$)
Vogel et al. (2016)	BSi	FTIRS	Marine sediments, atlantic ocean. High applicability of a local model ($R^2 = 0.97$), good transferability from older model ($R^2 = 0.87$)

NIRS near infrared reflectance spectroscopy, *FTIRS* Fourier-transformed infrared reflectance spectroscopy, *TOC* total organic carbon, *TN* total nitrogen, *TP* total phosphorus, *LOI* loss on ignition, *TIC* total inorganic carbon, *BSi* biogenic silica

(Alm 1993). For one lake, Uhca Rohči 1, the lithology is provided in supplementary material (ESM 1).

NIRS measurements

Approximately 1 g of wet sediment was dried at 50 °C for 24 h, ground in a mortar and scanned with a portable spectrometer (Fieldspec 3, ASD Inc, Boulder, CO). Spectra were measured as reflectance in the 350–2500 nm spectral range with resolution of 1.4 nm in the 250–1050 nm region and 2 nm in the 1000–2500 nm regions, and automatically interpolated to 1-nm resolution. Each sample was scanned five times in a black polyacetal sample holder, rotating and mixing the samples between each scan to incorporate all spectral variability for each sample. The repeated spectra were averaged into a single spectrum per sample. On average, approximately 500 samples could be scanned with the NIRS method per week.

Table 2 Summary of LOI values for each lake sediment record included in the NIRS models

Lake	Zone	Latitude	Longitude	Lithology	n	Min	Median	Mean	Max
Øvre Æråsvatnet (Andøya)	Boreal	69.25° N	16.05° E	Silty gyttja with sand laminae	86	5.1	30.7	42.2	73.9
Uhca Rohči 1 (Varanger)	Sub-arctic	70.32° N	30.02° E	Organic silt to gyttja	62	17.1	59.7	56.5	84.9
Uhca Rohči 2 (Varanger)	Sub-arctic	70.32° N	30.03° E	Silty clay to detrital silty-clay gyttja	78	15.1	29.2	30.3	38.3
Gauptjern (Troms)	Boreal	65.85° N	19.62° E	Silty gyttja to brownish detrital gyttja	136	11.9	51.4	52.6	83.3
Skartjørna (Svalbard)	High arctic	77.96° N	13.82° E	Clayey and silty laminae	40	4.5	6.0	6.8	10.5
Bolshoye Schuchye (Polar Urals)	Sub-arctic	67.87° N	66.31° E	Minerogenic silts with sand laminae	135	1.0	3.2	3.5	5.2

Sample size (n), minimum (min) and maximum (max) LOI values are given in percentage

LOI

Reference LOI values were obtained following the method by Dean ([1974](#)): drying samples at 105 °C overnight and weighed, followed by combustion in ceramic crucibles at 550 °C for 2–4 h, then re-weighed. LOI was calculated as:

$$LOI(\%) = \frac{Dry\ weight\,(g) - 550C\ remaining\ weight\,(g)}{Dry\ weight\,(g)} * 100$$

Statistical analyses were performed in R 3.2.2 (R Development Core Team [2014](#)) using partial least squares regression (Martens and Næs [1989](#)) contained in the "pls" package (Mevik and Wehrens [2007](#)). Data pre-processing tools i.e. derivatives, smoothing and spectra standardization were applied from the "pro-spectr" package (Stevens and Ramirez–Lopez [2014](#)). The following data transformations were tested for the model development: centering, scaling, smoothing based on moving averages, standard normal variate and 1st and 2nd order derivatives (Stevens and Ramirez–Lopez [2014](#)).

Seven individual models were developed for LOI. The first model included all the samples from the six sediment cores (n = 537): calibration and validation sets were created using the Kennard-Stone algorithm (Kennard and Stone [1969](#)) to ensure a proper spectral variability between calibration and validation, where 85% of the total sample was assigned to the calibration dataset and 15% was assigned to the validation dataset. The remaining six models were developed by subtracting one lake from the calibration dataset and validating the resulting model against this record (Table [2](#)). All the models were internally cross-validated with a 10-fold cross validation.

The most parsimonious models were selected based on a high coefficient of determination (R^2), given a number of latent variables (k) and low root mean squared error of the cross-validation (RMSECV), which assesses the error between NIRS measured and reference values. Finally, each calibration model was tested against its respective validation set: coefficient of determination (R^2), root mean square error of the predictions (RMSEP), bias (systematic error) and the intercept and slope of the linear fit of the predictions were calculated to assess the applicability of the model.

Intercept and slope of the linear fit were used to apply corrections based on the equation y = mx + b, where y is the corrected LOI value, m is the slope, b is the intercept and x is the NIRS measured raw value.

Results

LOI values ranged from very low to very high (1.03% to 85%), with a high overlap observed in the range of values between different lakes (Table [2](#)). The dataset covers a large variation in sediment composition, with samples ranging from organic-rich gyttja to
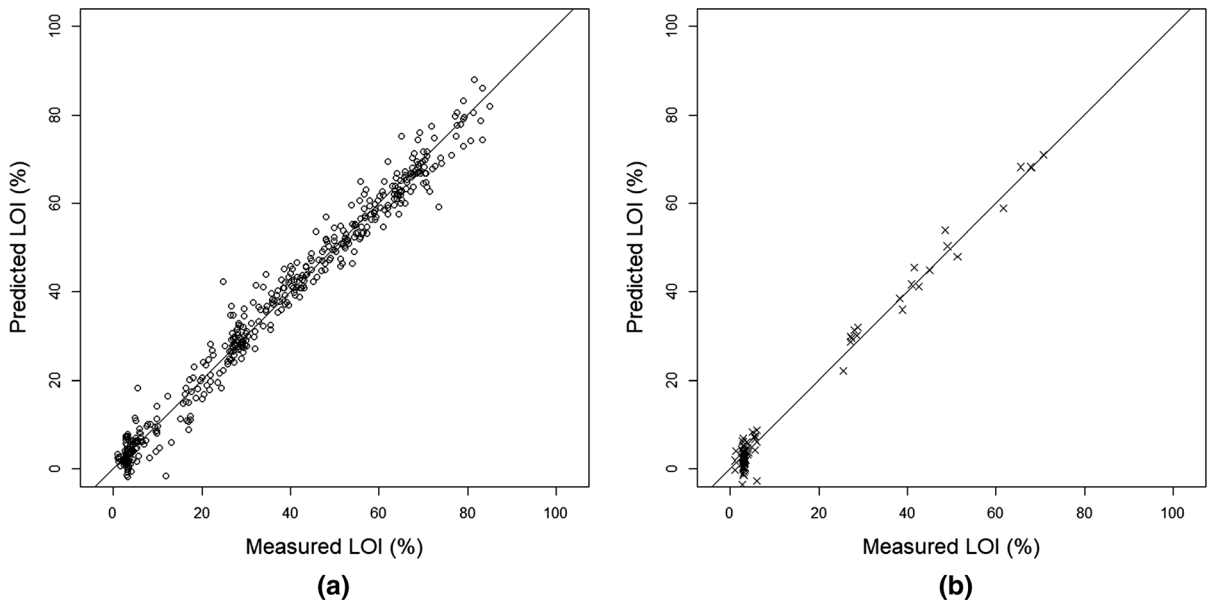
Fig. 1 **a** Calibration and **b** validation of the full LOI dataset including 537 samples from six lakes

Table 3 Calibration parameters of the NIRS models for LOI prediction

	Model	Full	Without Øvre Æråsvatnet	Without Uhca Rohči1	Without Uhca Rohči2	Without Gauptjern	Without Polar Urals	Without Skartjørna
	Data pre-treatment	SNV, derivative 1–2–23	SNV, derivative 0–2–23	SNV, derivative 0–2–23	SNV, derivative 0–2–23	SNV, derivative 0–2–23	SNV, derivative 0–2–23	SNV, derivative 0–2–23
	k	15	15	15	15	15	15	15
Calibration	R^2	0.98	0.96	0.97	0.98	0.97	0.95	0.96
	RMSECV	3.3	4.79	4.33	3.43	3.90	4.46	4.64
Validation	R^2	0.99	0.92	0.87	0.95	0.82	0.22	0.17
	RMSEP	2.46	5.47	9.34	19.31	7.64	17.95	7.49
	Bias	0.11	0.83	− 8.66	16.99	1.93	13.69	5.47
	Intercept	− 0.39	6.78	7.07	11.32	− 2.46	12.3	− 7.77
	Slope	1.02	0.82	1.05	0.5	1.01	− 7.21	1.34
Validation (Slope corrected)	RMSEP		5.27	3.3	4.22	7.31	1.37	4.26
	Bias		0	− 0.07	0.16	0	0	1.9

Standard normal variate (SNV), and the numbers after the derivative stand for differentiation order, polynomial order and window size respectively, number of latent variables (k), coefficient of determination between model fitted and reference LOI measurements (R^2), RMSECV and RMSEP are Root Mean Squared Error of the Cross Validation and Prediction, respectively, bias is the systematic error between reference and NIRS-measured values and Intercept and Slope represent the coefficients of the linear fit between the reference and NIRS-measured values
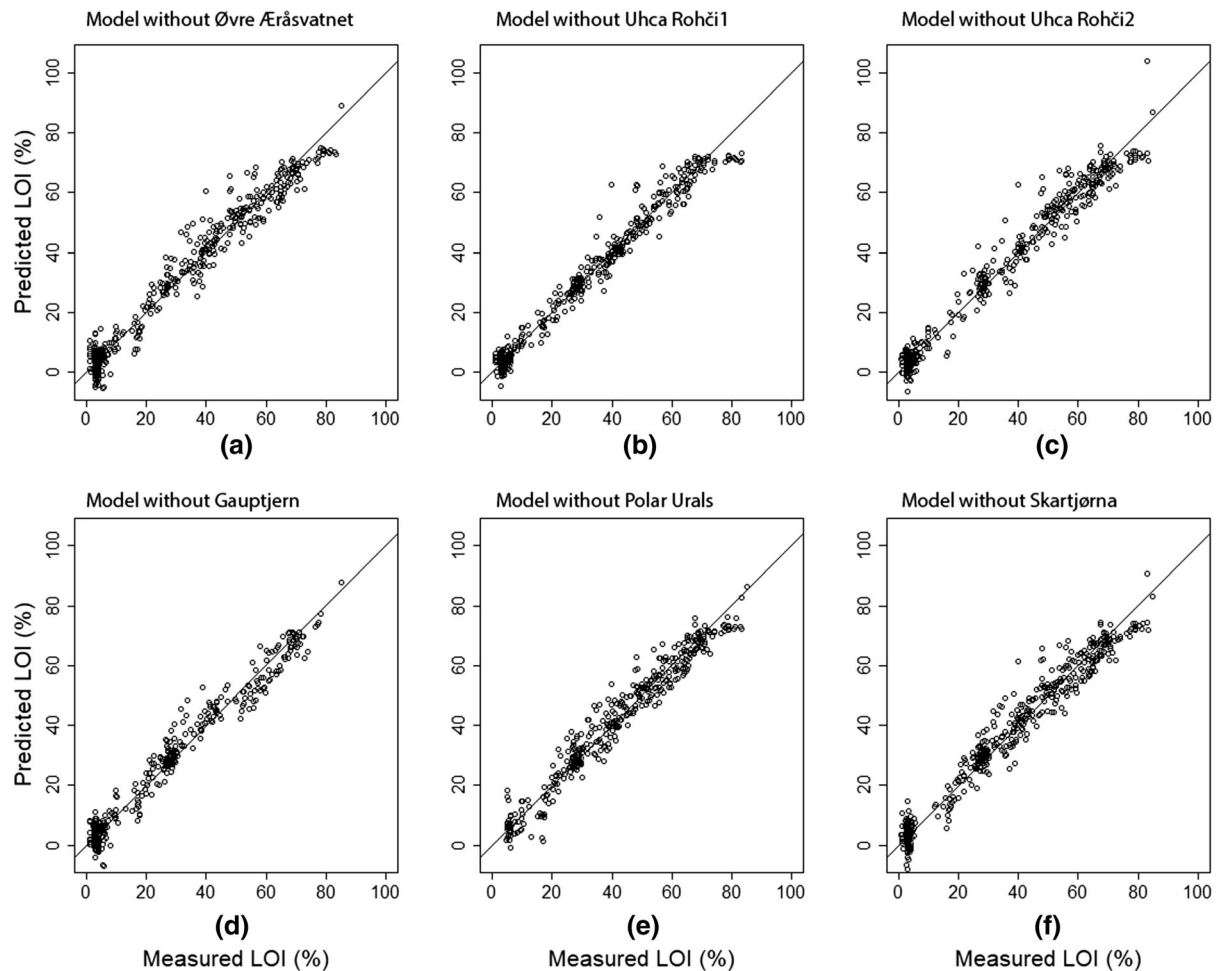
Fig. 2 Calibration plots for the LOI models when substracting a lake from the database. The solid line represents the perfect fit (1:1) line. Figures represent the models excluding (**a**) Øvre Æråsvatnet (n_{cal} = 434), (**b**) Uhca Rohči 1 (n_{cal} = 458), (**c**) Uhca Rohči 2 (n_{cal} = 442), (**d**) Gauptjern (n_{cal} = 401), (**e**) Polar Urals (n_{cal} = 402) and (**f**) Skartjørna (n_{cal} = 497)

predominantly minerogenic silts and clay with and without a significant $CaCO_3$ content (Table 2).

The full model including all the samples from the six sediment cores performed best (Fig. 1), with good fit in both the calibration and internal validation datasets (Table 3). The remaining models showed good model performance on the calibrations (Fig. 2) but needed intercept and slope correction on the external validations (Fig. 3, Table 3). In addition, the model excluding Gauptjern (Fig. 3d) showed a curvilinear response in the highest LOI values, most likely due to high $CaCO_3$ content (Jensen and Vorren 2008). Once corrected for intercept and slope, RMSEP values were reduced to similar levels as the full model

(Table 3), showing that intercept and slope corrections result in highly precise estimates of sediment LOI on samples that do not belong to the reference population.

The samples belonging to the polar Urals and Skartjørna showed a poor coefficient of determination on the validation set (R^2 of 0.22 and 0.17, respectively) (Fig. 3e, f), although RMSEP values were similar to the other models after corrected for intercept and slope (RMSEP 1.37 and 4.26, respectively).
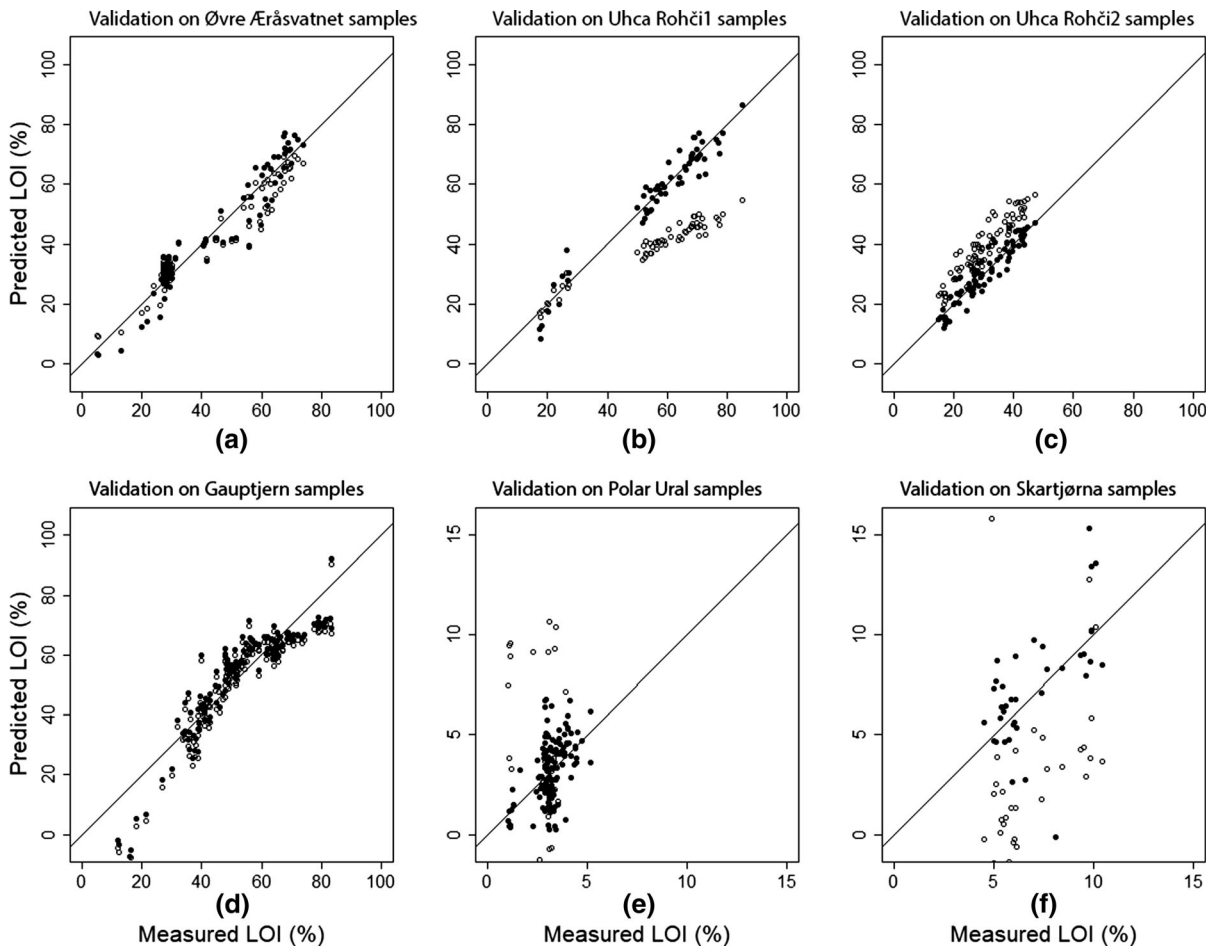
Fig. 3 Validation plots for the LOI models when substracting a lake from the database. The solid line represents the perfect fit (1:1) line. Hollow circles represent uncorrected predictions and solid black circles represent intercept and slope corrected predictions. Figures represent validation of the excluded lakes from their respective models (**a**) Øvre Æråsvatnet ($n_{val} = 86$), (**b**) Uhca Rohči 1 ($n_{val} = 62$), (**c**) Uhca Rohči 2 ($n_{val} = 78$), (**d**) Gauptjern samples ($n_{val} = 119$), (**e**) Polar Urals ($n_{val} = 135$) and (**f**) Skartjørna ($n_{val} = 40$)

Discussion

The high predictive ability of LOI based on NIRS reported here extends previous studies showing NIRS as a reliable, non-destructive method for measuring lake sediment properties, both for within-site samples (Stenberg et al. 2010; Gholizadeh et al. 2013) or samples from contrasting sites that are not part of the calibration model (Rosén and Persson 2006; Rosén et al. 2011; Meyer-Jacob et al. 2017). A single NIRS spectrum can be used to simultaneously determine several sediment properties with no increased costs: one operation for sample preparation and scan is enough to estimate several variables, if predictive models are established (Malley et al. 2000; Cozzolino and Morón 2006). Cost per sample is further reduced with a greater number of predictive models available: sediment samples scanned in previous studies can retrospectively be analysed through their NIRS spectra and a new dimension added into the data gathering even after the samples have been scanned and analyzed with destructive methods. An added advantage is that, due to the small amount of sediment required to acquire a spectrum (approximately 1 g), high-resolution down-core analyses can be performed.
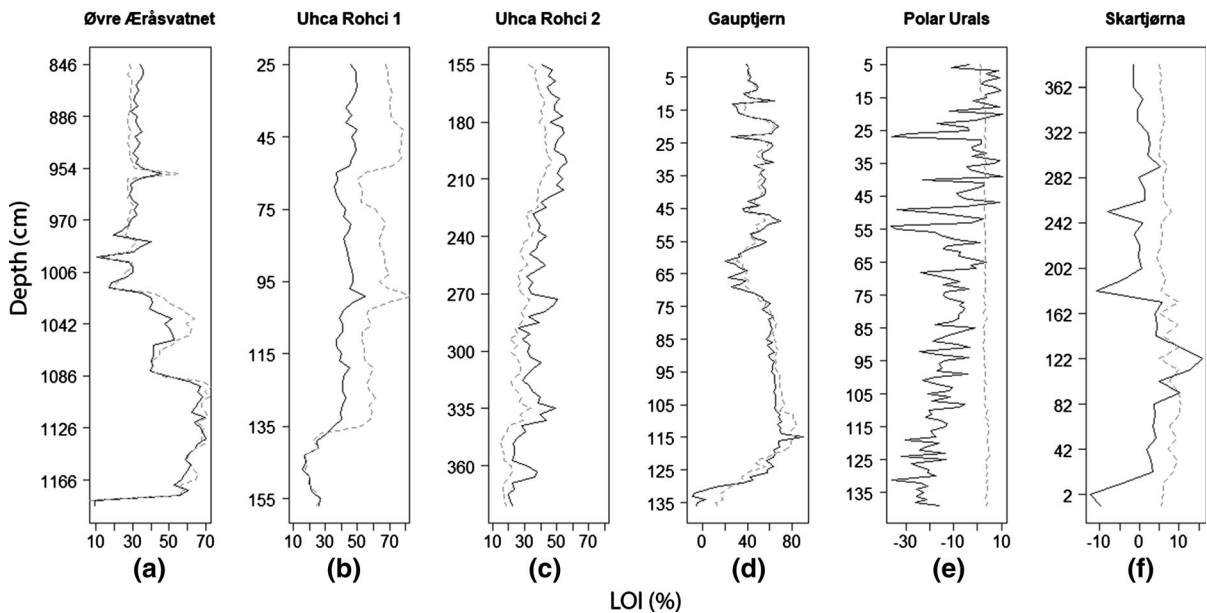
Fig. 4 LOI on the cores from (**a**) Øvre Æråsvatnet, (**b**) Uhca Rohči 1, (**c**) Uhca Rohči 2, (**d**) Gauptjern, (**e**) Polar Ural and (**f**) Skartjørna lakes. Gray dashed line represents reference LOI values, solid black line represents NIRS predicted raw values (uncorrected)

While it is expected that NIRS predictions are most accurate when limited to samples with similar properties to those included in the calibration dataset (Foley et al. 1998; Chodak 2008), our study shows that comprehensive models including enough variability in stratigraphy or geographical origin can be applicable to other samples. Thus, we suggest that this is a first step towards creating worldwide lacustrine spectral libraries to develop true global models applied to lacustrine sediments (Viscarra Rossel 2009; Rosén et al. 2011; Stenberg and Viscarra Rossel 2016).

When sequentially removing a lake from the calibration set and then testing the model against it, we found a linear bias in most of the lakes. After performing the linear corrections, we obtained highly accurate estimates ($R^2 > 0.82$, RMSEP < 7.5, except for the polar Ural and Skartjørna samples), comparable to those of the model including all the lakes ($R^2 = 0.98$, RMECV = 3.3). The polar Ural and Skartjørna samples showed a poor coefficient of determination, but similar RMSEP values to the other models after intercept and slope corrections. Given the low variability in the LOI values on these two cores, the apparent model performance seems to be suboptimal: such cases with consistently low values along the core will raise concerns about the applicability of the model. However, the NIRS-measured values

reveal similar patterns in LOI between the raw predictions and the measured LOI values on the core (Fig. 4), although apparent performance may seem worse, the general LOI pattern along the core is correctly detected and the RMSEP shows that the low R^2 values are an effect of the small LOI range in these two cores.

The prediction of the Gauptjern lake (Fig. 3d) showed a non-linear fit towards high LOI values. This is expected when core properties are out of the predictive ability of the model, due to a number of parameters such as chemical composition (e.g. $CaCO_3$ content) or particle size distribution (Barthes et al. 2006). The use of a validation subset for each core along the gradient of chemical/physical properties of the sediment (e.g. organic matter, carbon content) is a safe way to identify such issues. Once the problematic region has been detected, it can be corrected for linear bias, or the core (or core section) analyzed with traditional measurements, to incorporate it into the model. Even raw (uncorrected) LOI predictions from NIRS models already provide a first assessment of the LOI patterns down the core (Fig. 4). This helps to focus more intensive analyses based on other approaches in core regions with interesting patterns such as steep changes in LOI, or unusual values.

To establish a robust validation of NIRS models in the future, McTiernan et al. (1998) recommend a minimum sample size of 20 samples from a single lake to develop a predictive model. However, we suggest to scan a higher amount (minimum of 40 to 50) of samples for each lake, to ensure representativeness of all intrinsic properties that each sediment origin presents. Robust models require several hundreds of samples representing a large range of LOI: once the predictive model is established, we suggest measuring a subset of samples (approx. 20% of the original number of samples) using the classical LOI methodology, selecting samples according to their NIRS predicted value to ensure a proper spread along the LOI values. In addition, core sections with new properties (stratigraphy, grain size, etc.) need to be more intensively sampled and included in the model afterwards. This will result in accurate correction factors (i.e. intercept and slope) for high-quality NIRS-inferred LOI estimates (Table 3). This process needs to be done for each new sediment core analysed by NIRS. It is expected that incorporating these validation samples into the model will improve the model robustness and increase the applicability for different samples from different lakes, resulting in a synergistic effect that will reduce the future need for intercept and slope corrections.

Conclusions

This study shows that NIRS can be used to estimate LOI in a wide variety of lake sediment types from six geographically distinct lakes with only minimal calibration samples. NIRS therefore has the potential to become a new standard procedure in lacustrine sediment research for the simultaneous and high resolution measurement of several sediment properties with only a 20% subset of validation samples needed to be analyzed with traditional methods in order to adjust the NIRS measured values, thus saving time and costs in sediment analyses. Such transferable models are especially valuable when large sets of samples (i.e. several cores from a lake, or long cores) are to be analyzed. Future work should focus on adding samples with different stratigraphic properties and geographical regions to build a more robust library.

References

Alm T (1993) Øvre AEråsvatn—palynostratigraphy of a 22,000 to 10,000 BP lacustrine record on Andøya, northern Norway. Boreas 22:171–188. https://doi.org/10.1111/j.1502-3885.1993.tb00178.x

Alsos IG, Sjögren P, Edwards ME, Landvik JY, Gielly L, Forwick M, Coissac E, Brown AG, Jakobsen LV, Foreid MK, Pedersen MW (2016) Sedimentary ancient DNA from Lake Skartjørna, Svalbard: assessing the resilience of arctic flora to Holocene climate change. The Holocene 26:627–642. https://doi.org/10.1177/0959683615612563

Balsam WL, Deaton BC (1996) Determining the composition of late quaternary marine sediments from NUV, VIS, and NIR diffuse reflectance spectra. Mar Geol 134:31–55. https://doi.org/10.1016/0025-3227(96)00037-0

Barthes BG, Brunet D, Ferrer H, Chotte JL, Feller C (2006) Determination of total carbon and nitrogen content in a range of tropical soils using near infrared spectroscopy: influence of replication and sample grinding and drying. J Near Infrared Spectrosc 14:341–348. https://doi.org/10.1255/jnirs.686

Birks HH, Birks HJB (2006) Multi-proxy studies in palaeolimnology. Veg Hist Archaeobot 15:235–251. https://doi.org/10.1007/s00334-006-0066-6

Björck S, Håkansson H, Zale R, Karlen W, Jonsson BL (1991) A late Holocene lake sediment sequence from Livingston

Island, South Shetland Islands, with palaeoclimatic implications. Antarct Sci 3:61–72. https://doi.org/10.1017/S095410209100010X

Chodak M (2008) Application of near infrared spectroscopy for analysis of soils, litter and plant materials. Pol J Environ Stud 17:631–642

Clarke CL, Edwards ME, Brown AG, Gielly L, Lammers Y, Heintzman PD, Ancin-Murguzur FJ, Bråthen K-A, Goslar T, Alsos IG (2019) Holocene floristic diversity and richness in northeast Norway revealed by sedimentary ancient DNA (seda DNA) and pollen. Boreas 48:299–316. https://doi.org/10.1111/bor.12357

Cozzolino D, Morón A (2003) The potential of near-infrared reflectance spectroscopy to analyse soil chemical and physical characteristics. J Agric Sci 140:S0021859602002836. https://doi.org/10.1017/S0021859602002836

Cozzolino D, Morón A (2006) Potential of near-infrared reflectance spectroscopy and chemometrics to predict soil organic carbon fractions. Soil Tillage Res 85:78–85. https://doi.org/10.1016/j.still.2004.12.006

Dean WE (1974) Determination of carbonate and organic matter in calcareous sediments and sedimentary rocks by loss on ignition: comparison with other methods. J Sediment Petrol 44:242–248

Foley WJ, McIlwee A, Lawler I, Aragones L, Woolnough AP, Berding N (1998) Ecological applications of near infrared reflectance spectroscopy—a tool for rapid, cost-effective prediction of the composition of plant and animal tissues and aspects of animal performance. Oecologia 116:293–305. https://doi.org/10.1007/s004420050591

Gholizadeh A, Luboš B, Saberioon M, Vašát R (2013) Visible, near-infrared, and mid-infrared spectroscopy applications for soil assessment with emphasis on soil organic matter content and quality: state-of-the-art and key issues. Appl Spectrosc 67:1349–1362. https://doi.org/10.1366/13-07288

Hahn A, Kliem P, Ohlendorf C, Zolitschka B, Rosén P (2013) Climate induced changes as registered in inorganic and organic sediment components from Laguna Potrok Aike (Argentina) during the past 51ka. Quat Sci Rev 71:154–166. https://doi.org/10.1016/j.quascirev.2012.09.015

Heanes DL (1984) Determination of total organic C in soils by an improved chromic acid digestion and spectrophotometric procedure. Commun Soil Sci Plant Anal 15:1191–1213. https://doi.org/10.1080/00103628409367551

Heiri O, Lotter AF, Lemcke G (2001) Loss on ignition as a method for estimating organic and carbonate content in sediments: reproducibility and comparability of results. J Paleolimnol 25:101–110. https://doi.org/10.1023/A:1008119611481

Inagaki T, Shinozuka Y, Yamada K, Yonenobu H, Hayashida A, Tsuchikawa S, Yoshida A, Hoshino Y, Gotanda K, Yasuda Y (2012) Rapid prediction of past climate condition from lake sediments by near-infrared (NIR) spectroscopy. Appl Spectrosc 66:673–679. https://doi.org/10.1366/11-06418

Jensen C, Vorren K-D (2008) Holocene vegetation and climate dynamics of the boreal alpine ecotone of northwestern Fennoscandia. J Quat Sci 23:719–743. https://doi.org/10.1002/jqs.1155

Kaye W (1954) Near-infrared spectroscopy: spectral identification and analytical applications. Spectrochim Acta 6:257–287

Kaye W (1955) Near infrared spectroscopy: instrumentation and technique. Spectrochim Acta 7:181–204

Kennard R, Stone L (1969) Computer aided design of experiments. Technometrics 11:137–148

Korsman T, Nilsson MB, Landgren K, Renberg I (1999) Spatial variability in surface sediment composition characterised by near-infrared (NIR) reflectance spectroscopy. J Paleolimnol 21:61–71. https://doi.org/10.1023/A:1008027001745

Leach CJ, Wagner T, Jones M, Juggins S, Stevenson AC (2008) Rapid determination of total organic carbon concentration in marine sediments using Fourier transform near-infrared spectroscopy (FT-NIRS). Org Geochem 39:910–914. https://doi.org/10.1016/j.orggeochem.2008.04.012

Malley DF, Williams PC, Stainton MP (1996) Rapid measurement of suspended C, N, and P from precambrian shield lakes using near-infrared reflectance spectroscopy. Water Res 30:1325–1332

Malley DF, Rönicke H, Findlay DL, Zippel B (1999) Feasibility of using near-infrared reflectance spectroscopy for the analysis of C, N, P, and diatoms in lake sediments. J Paleolimnol 21:295–306. https://doi.org/10.1023/A:1008013427084

Malley DF, Lockhart L, Wilkinson P, Hauser B (2000) Determination of carbon, carbonate, nitrogen, and phosphorus in freshwater sediments by near-infrared reflectance spectroscopy: rapid analysis and a check on conventional analytical methods. J Paleolimnol 24:415–425. https://doi.org/10.1023/A:1008151421747

Martens H, Naes T (1989) Multivariate calibration. John Wiley & Sons

McTiernan KB, Garnett MH, Mauquoy D, Ineson P, Couteaux MM (1998) Use of near-infrared reflectance spectroscopy (NIRS) in palaeoecological studies of peat. Holocene 8:729–740. https://doi.org/10.1191/095968398673885510

Mecozzi M, Pietrantonio E, Amici M, Romanelli G (2001) Determination of carbonate in marine solid samples by FTIR-ATR spectroscopy. Analyst 126:144–146. https://doi.org/10.1039/b009031j

Mevik B-H, Wehrens R (2007) The pls package: principal component and partial least squares regression in R. J Stat Softw 18:1–24. https://doi.org/10.1002/wics.10

Meyer-Jacob C, Vogel H, Gebhardt AC, Wennrich V, Melles M, Rosen P (2014) Biogeochemical variability during the past 3.6 million years recorded by FTIR spectroscopy in the sediment record of Lake El'gygytgyn. Far East Russ Arct Clim Past 10:209–220. https://doi.org/10.5194/cp-10-209-2014

Meyer-Jacob C, Michelutti N, Paterson AM, Monteith D, Yang H, Weckström J, Smol JP, Bindler R (2017) Inferring past trends in lake water organic carbon concentrations in northern lakes using sediment spectroscopy. Environ Sci Technol 51:13248–13255. https://doi.org/10.1021/acs.est.7b03147

Nduwamungu C, Ziadi N, Parent L-E, Tremblay GF, Thuriès L (2009) Opportunities for, and limitations of, near infrared

reflectance spectroscopy applications in soil analysis: a review. Can J Soil Sci 89:531–541. https://doi.org/10.4141/CJSS08076

Nesje A, Dahl SO (2001) The Greenland 8200 cal. yr BP event detected in loss-on-ignition profiles in Norwegian lacustrine sediment sequences. J Quat Sci 16:155–166. https://doi.org/10.1002/jqs.567

Pearson EJ, Juggins S, Tyler J (2014) Ultrahigh resolution total organic carbon analysis using Fourier transform near infrared reflectance spectroscopy (FT-NIRS). Geochem Geophys Geosyst 15:292–301. https://doi.org/10.1002/2013GC004928

R Development Core Team (2014) R: a language and environment for statistical computing. R Foundation for Statistical Computing, Vienna, Austria. http://www.R-project.org/. R Found Stat Comput Vienna, Austria

Roggo Y, Chalus P, Maurer L, Lema-Martinez C, Edmond A, Jent N (2007) A review of near infrared spectroscopy and chemometrics in pharmaceutical technologies. J Pharm Biomed Anal 44:683–700. https://doi.org/10.1016/j.jpba.2007.03.023

Rosén P, Persson P (2006) Fourier-transform infrared spectroscopy (FTIRS), a new method to infer past changes in tree-line position and TOC using lake sediment. J Paleolimnol 35:913–923. https://doi.org/10.1007/s10933-005-5010-8

Rosén P, Vogel H, Cunningham L, Reuss N, Conley DJ, Persson P (2010) Fourier transform infrared spectroscopy, a new method for rapid determination of total organic and inorganic carbon and biogenic silica concentration in lake sediments. J Paleolimnol 43:247–259. https://doi.org/10.1007/s10933-009-9329-4

Rosén P, Vogel H, Cunningham L, Hahn A, Hausmann S, Pienitz R, Zolitschka B, Wagner B, Persson P (2011) Universally applicable model for the quantitative determination of lake sediment composition using fourier transform infrared spectroscopy. Environ Sci Technol 45:8858–8865. https://doi.org/10.1021/es200203z

Stenberg B, Viscarra Rossel R (2016) Guest editorial: near infrared spectroscopy for a better understanding of soil.

J Near Infrared Spectrosc. https://doi.org/10.1255/jnirs.1234

Stenberg B, Rossel RV, Mouazen M, Wetterlind J (2010) Visible and near infrared spectroscopy in soil science. Adv Agron 107(107):163–215. https://doi.org/10.1016/s0065-2113(10)07005-7

Stevens A, Ramirez-Lopez L (2014) An introduction to the prospectr package. R package Vignette, Report No.: R Package Version 0.1.3

Svendsen JI, Faerseth LMB, Gyllencreutz R, Haflidason H, Henriksen M, Hovland MN, Lohne ØS, Mangerud J, Nazarov D, Regnéll C, Schaefer JM (2018) Glacial and environmental changes over the last 60,000 years in the Polar Ural Mountains, Arctic Russia, inferred from a high-resolution lake record and other observations from adjacent areas. Boreas. https://doi.org/10.1111/bor.12356

Viscarra Rossel R (2009) The soil spectroscopy group and the development of a global soil spectral library. NIR News 20:14. https://doi.org/10.1255/nirn.1131

Vogel H, Rosén P, Wagner B, Melles M, Persson P (2008) Fourier transform infrared spectroscopy, a new cost-effective tool for quantitative analysis of biogeochemical properties in long sediment records. J Paleolimnol 40:689–702. https://doi.org/10.1007/s10933-008-9193-7

Vogel H, Meyer-Jacob C, Thöle L, Lippold JA, Jaccard SL (2016) Quantification of biogenic silica by means of Fourier transform infrared spectroscopy (FTIRS) in marine sediments. Limnol Oceanogr Methods 14:828–838. https://doi.org/10.1002/lom3.10129

Wang J, Zhu L, Wang Y, Gao S, Daut G (2012) A comparison of different methods for determining the organic and inorganic carbon content of lake sediment from two lakes on the Tibetan Plateau. Quat Int 250:49–54. https://doi.org/10.1016/j.quaint.2011.06.030