Citation for the published paper:
Wetterlind, J., Stenberg, B. and Jonsson, A. (2008) Near infrared reflectance spectroscopy compared with soil clay and organic matter content for estimating within-field variation in N uptake in cereals. *Plant and soil*. Volume: 302 Number: 1-2, pp. 317-327.
http://dx.doi.org/10.1007/s11104-007-9489-9

Near infrared reflectance spectroscopy compared with soil clay and organic matter content for estimating within-field variation in N uptake in cereals

Johanna Wetterlind, Bo Stenberg and Anders Jonsson
Division of Precision Agriculture, Department of Soil Science, SLU, P.O. Box 234, SE-532 23 Skara, Sweden.

Key words: NIR, nitrogen mineralisation, precision agriculture

Abstract

Adjusting fertiliser applications to within-field variations in nitrogen (N) mineralisation during the growing season can increase yields, improve crop quality, reduce costs and decrease nutrient losses to the environment. Predicting such variations at a reasonable cost is therefore important. In a three-year study, Near Infrared Reflectance (NIR) spectroscopy was compared with soil organic matter (SOM) and clay content as predictors of plant N uptake using cross-validated PLS (Partial Least Squares) regression models. Plant N uptake was measured as total nitrogen in aboveground plant parts at harvest, in plots without N fertilisation within three different fields in southern Sweden. NIR spectroscopy and combined clay and SOM content resulted in equally good estimations of plant N uptake in fields with large variation in SOM content. Cross-validated NIR calibrations for plant N uptake within fields for separate years resulted in r^2 values of 0.75-0.85 and average cross-validation errors of 11-16 kg N ha^{-1} for two fields (one year excluded at one field because of farmyard manure application). No significant improvements were seen when NIR-spectra, clay and SOM were included in the same model, suggesting that the additional predictive capacity of NIR over SOM relates to soil texture variations. NIR calibrations also performed poorly in one field where plant N uptake could not be explained by SOM or clay content. Predictions within fields between years produced r^2 values of 0.54-0.92 and prediction errors of 8-20 kg N ha^{-1} for one field. These results confirm that N uptake prediction accuracy can be improved by using NIR spectroscopy in fields with large SOM variations. However, good estimations could not be made between fields, indicating difficulties in creating more general calibration models for large geographical areas.

Introduction

Considerable amounts of N can be mineralised during the growing season and the variation between and within fields can be significant (Börjesson et al. 1999; Delin and Lindén 2002). Adjusting fertiliser applications with respect to this variation can therefore both reduce costs and decrease nutrient losses to the environment. In precision agriculture, global positioning system (GPS) technology and the development of geographical information systems (GIS) together with variable rate technology (VRT) have made it possible for farmers to adjust crop inputs as they drive through the fields. This has increased the demand for information at a high spatial resolution. However, increasing the amount of conventional methods of laboratory analyses to meet these demands fast becomes too costly and time-consuming.

NIR (Near Infrared Reflectance) techniques are fast and non-destructive. They require only limited sample preparation and the potential exists to develop instruments for online field measurements (Mouazen et al. 2005). NIR spectra have been shown to estimate total carbon or soil organic matter (SOM) content in a number of studies from different parts of the world (Bendor and Banin 1995; Chang et al. 2001; Cozzolino and Moron 2006; Islam et al. 2003; Shepherd and Walsh 2002). NIR spectra have also been related to other soil properties that can be expected to influence N mineralisation, such as the quality of SOM (Hartmann and Appel 2006; Reeves et al. 2006; Terhoeven-Urselmans et al. 2006) and soil texture (Chang et al. 2001; Sorensen and Dalsgaard 2005; Stenberg et al. 1995). NIR spectroscopy has also been used with promising results to determine both potentially mineralisable N, measured as accumulated mineral N in aerobic or anaerobic incubations (Chang et al. 2001; Dunn et al. 2000; Fystro 2002; Shepherd and Walsh 2002), as well as N uptake in crops within and between fields (Börjesson et al. 1999; Dunn et al. 2000; Stenberg et al. 2005).

Börjesson et al. (1999) suggested that models based on NIR spectra could indirectly take into account the water supply function of a soil through e.g. its texture. Stenberg et al. (2005) found that even though within-field variation in SOM constituted a large part of the NIR spectra for predicting N uptake, NIR spectra included more relevant information and thus performed better. The fields in the studies by Börjesson et al. (1999) and Stenberg et al. (2005) had high variation in SOM and further investigations are needed to study the possibility of obtaining equally accurate results in fields with lower SOM content. Even though Stenberg et al. (2005) included two different fields in their investigation, these fields were located in the same river basin. Thus, more investigations are also needed to study the potential for making equally good calibrations in other geographical areas with different soil types.

The aims of this work were (i) to compare predictions of plant-available N, measured as plant N uptake at harvest in plots without N fertilisation, using calibrations of NIR soil spectra with calibrations of SOM and clay content (ii) to include fields without large variations in SOM content and (iii) to further investigate the possibility of making one calibration model that could be used on other fields with somewhat different climatic conditions and parent material.

Materials and methods

Sites and experimental design

A field study was conducted at three fields in the south of Sweden located at the farms Ribbingsberg (58°06´N, 12°50´E), Nybble (59°13´N, 15°0´E) and Hacksta (59°33´N, 17°02´E). Annual average precipitation (standard 30-year average) is approx. 700 mm at Ribbingsberg, 600 mm at Nybble and 500 mm at Hacksta (Alexandersson and Eggertsson Karlström 2001). The size of the fields was 15 ha at Ribbingsberg, 11 ha at Nybble and 25 ha at Hacksta. The fields at Nybble and Ribbingsberg were selected for their large variation in SOM content, as apparent from farm soil maps and previous investigations, whereas the field at Hacksta had a variation in SOM content more typical for farms in southern Sweden. The soils at Nybble and Hacksta were on average silty clays with variations from clay to silty loam. Ribbingsberg was on average a loam, but varied from silty clay to loamy sand. The study was carried out during the three growing seasons 2003-2005 at Ribbingsberg and Nybble and during 2004 at Hacksta. The crop rotation at all three fields was dominated by cereals and cereals were grown in all fields during the experiment and in the preceding year. At Nybble the crop was oats in 2003-2004 and spring barley in 2005. At Ribbingsberg winter wheat was grown in 2003 and 2005 and oats in 2004. The crop at Hacksta was winter wheat. No farmyard manure was used at Nybble or Hacksta. At Ribbingsberg, however, 30 ton pig slurry had been applied to the field in autumn 2002. Pesticides and herbicides were used according to local advisory service recommendations and regional practices.

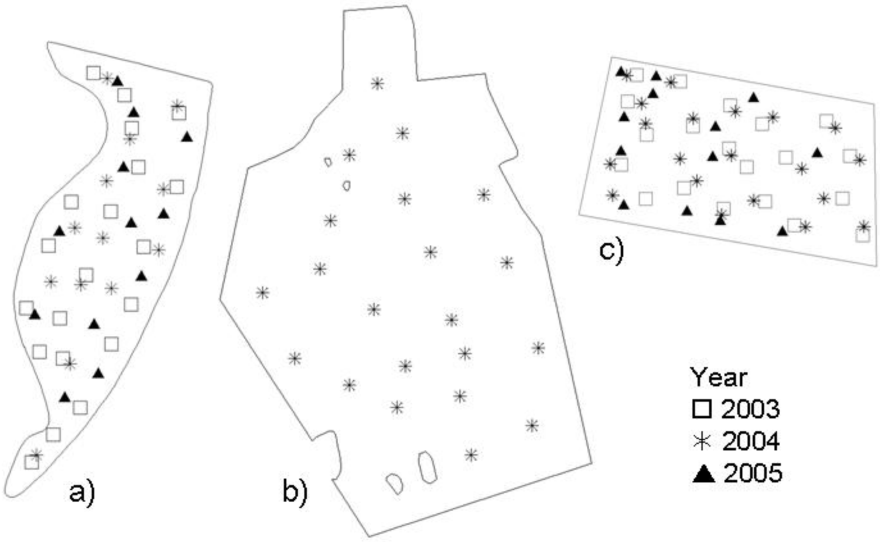


Figure 1. Location of unfertilised plots during the years 2003 to 2005 at a) Ribbingsberg, b) Hacksta and c) Nybble.

During the growing season, 12 to 21 plots of approx. 24 m^2 in size with no nitrogen fertilisation (zero N-plots) were distributed over each field (Fig. 1). The plots were positioned with GPS with an accuracy between 0.5 and 2 m and visually distributed to cover within-field variation according to topography, clay content and SOM data from existing farm-soil mapping surveys. Each year the plots were relocated to avoid effects of no nitrogen fertilisation the previous year. Year two and three the plots were distributed to reduce gaps between plots from previous years and at the same time retain the variation.

Plant and soil analyses

At ripeness (growth state 91; Zadoks et al. 1974), all above-ground plant parts in four randomly distributed 0.25 m^2 quadrants were sampled and bulked together in each plot. The samples were dried at 60° C for 24 hours, threshed and analysed for total N in straw and grain using Dumas elemental analysis on a CNS-2000 analyser (LECO Corporation, St. Joseph, MI, USA). In the following, this is referred to as plant N uptake.

In April, soil samples were taken from three layers in each plot, 0-30 cm, 30-60 cm and 60-90 cm. Ten randomly distributed soil cores (30 mm in diameter) were taken from the topsoil in each plot and 8 cores (20 mm in diameter) from the two subsoil layers and bulked together to form one sample from each layer per plot. One exception was Nybble in 2003, when only soil from the 0-30 cm depth was collected. The soil samples were stored frozen, dried at 35 °C for two to three days and crushed to pass a 2 mm sieve before analyses.

SOM content was analysed as loss of ignition corrected for loss of structural water from clay minerals (Ekström 1927). For clay contents of 200 g kg^{-1} or lower, the correction factor is k = 0.1 x (% clay) and for clay contents higher than 200 g kg^{-1} k = 1.06 + 0.0047 x (% clay). Soil texture was divided into clay (>0.002 mm) silt (0.002-0.063 mm) and sand (0.063-2 mm). Clay content was analysed using a sedimentation method modified from Gee and Bauder (1986). Silt and sand fractions were determined by sieving. These conventionally analysed SOM and clay contents were used to predict plant N uptake in parallel to NIR.

NIR spectra were taken of each soil sample using a FieldSpec Pro FR scanning instrument (Analytical Spectral Devices Inc., Boulder, CO, USA, www.asdi.com). The instrument was equipped with a bare optic fibre connected to a probe with a 20 W Al-coated halogen tungsten light source positioned 7 cm over the sample. From each soil sample, reflectance spectra from two sub-samples were collected. Each spectrum consisted of 50 averaged sub-spectra from a rotating sample area of about 50 cm^2. Calibrations to an external white reference (Spectralon) were made between every two sub-samples. The range of measurement was 350-2500 nm in 1.4-2.0 nm intervals, with a spectral resolution of 3-10 nm. A wavelength interval of 1 nm was interpolated to the instrument output file. Wavelengths from the NIR region between 780 and 2500 nm were used in further calculations.

Data analyses

The NIR measurements were expressed as log reflectance^{-1} and the wavelength interval was averaged to every second nm. The two spectra from the sub-samples were averaged before further analysis. To enhance weak signals and reduce noise, each spectrum was transformed using the Savitzky-Golay technique with a first derivative before further analyses (Savitzky and Golay 1964). A second order polynomial was fitted to each spectrum in segments of five points. Before deciding on this transformation method, a number of first and second order derivatives as well as standard normal variate with and without de-trending (Barnes et al. 1989) were tested on a sub-set of the data.

Three sets of variables were compared for prediction of plant N uptake: i) NIR spectra, ii) traditionally analysed SOM and clay content; and iii) the combination of NIR spectra and traditionally analysed SOM and clay content. Plant N uptake was calibrated to the variables (with one exception) using the multivariate linear calibration technique PLS (Partial Least Squares) regression (Martens and Naes 1989). Calibration models were made both with data from the whole soil profile (0-90 cm) and with data from the 0-30 cm layer only. Calibrations with SOM and clay content from the 0-30 cm depth were made with multivariate linear regression (MLR) instead of PLS regression because of only two variables. In the calibration models with NIR spectra combined with traditionally analysed SOM and clay content, the impact of the NIR spectra with 860 wavelength variables compared to the two (0-30 cm) or six (0-90 cm) SOM and clay variables, would be to large. Therefore, a principal component analysis (PCA) was carried out for the NIR spectra and the first six principal components accounting for at least 90% of the variation were added to the SOM and clay content data as six new variables independent to each other.

Field specific calibration models were made. These were made for separate years as well as for combinations of years (12-52 samples). All calibration models were validated by full cross-validation through a leave-one-out procedure (Martens and Naes 1989). The calibration models were also used to predict plant N uptake between years within the same field and between fields within and between years.

To deal with possible problems with different levels of plant N uptake between the years due to different crops and weather conditions, each year's plant N uptake was also normalised by subtracting the mean and dividing by the standard deviation. Normalised plant N uptake was then calibrated to the same variable sets and in the same way as plant N uptake.

NIR spectra were also calibrated for SOM and clay content respectively. Two calibration models were made for each parameter and field, one calibration model including topsoil measurements and one calibration model including measurements from the two subsoil layers. Measurements from all the years' plots were included in both calibrations.

The calibration models were evaluated by the r^2 value between estimated and measured plant N uptake, by the root mean squared error of cross-validation (RMSECV) and prediction (RMSEP) and by ratio of performance to deviation (RPD) (standard deviation (s.d.) divided by RMSEP or RMSECV) (Williams 1987). For evaluation, the RPD values were divided into classes as suggested for soils by Malley et al. (2004) with RPD > 4 as excellent, RPD between 3 and 4 as successful, RPD between 2.25 and 3 as moderately successful and RPD between 1.75 and 2.25 as moderately useful. Data analysis and statistics regarding the calibrations and NIR-measurements were performed with Unscrambler® 9.0 (CAMO PROCESS AS, Oslo, Norway). For statistical comparison between the different methods, one-way ANOVA ($P<0.05$) was used (MINITAB version 14).

Results

Soil texture and SOM

Texture and SOM content in the three layers studied in the zero-N plots of all three field sites are summarised in Table 1. At Nybble, one of the plots in 2005 was excluded, as high clay content and exceptionally low SOM content in the topsoil indicated possible contamination by subsoil or mislabelling during sampling. The plot was also a strong outlier in the calibrations.

At Nybble and Hacksta, the highest SOM content was found in the topsoil, with decreasing amount and variation down the profile. At Ribbingsberg, however, the variation increased with depth and due to very high SOM content in a few plots, sometimes exceeding 400 g kg^{-1} at 60-90 cm depth, the average SOM content stayed more or less constant throughout the profile. The plots with the highest SOM content in the subsoil were located in a low part of the field close to a stream and had an increasing SOM content from the topsoil down to 90 cm

Table 1. Soil organic matter content and texture (g kg^{-1}) in (*n*) soil samples from three layers. In the topsoil, (*n*) equals the total number of plots for all years without nitrogen fertilisation

	0-30 cm					30-60 cm					60-90 cm				
	n	Mean	Min	Max	StDev	n	Mean	Min	Max	StDev	n	Mean	Min	Max	StDev
Nybble*															
SOM	52	80	32	191	36	30	27	<1	146	36	31	13	<1	62	16
Clay	52	349	120	620	108	30	440	200	620	118	31	460	280	670	110
Silt	52	600	360	820	110	30	530	370	770	120	31	510	320	690	100
Sand	52	48	20	90	15	30	20	10	60	10	31	30	10	160	30
Ribbingsberg**															
SOM	45	79	17	203	55	45	79	<1	340	100	45	71	<1	475	146
Clay	45	220	80	370	65	45	214	50	420	100	42	220	40	470	120
Silt	45	460	270	680	100	45	450	140	750	170	42	440	140	820	170
Sand	45	325	60	630	146	45	340	50	800	250	42	350	70	820	240
Hacksta															
SOM	20	36	20	56	11	20	7	<1	33	8	20	2	<1	22	5
Clay	20	402	230	520	80	20	500	250	630	100	20	540	220	770	160
Silt	20	440	350	540	50	20	410	320	570	60	20	380	210	540	80
Sand	20	154	50	380	82	20	100	20	290	88	20	80	10	260	90

* In 2003 only topsoil samples were collected. Two samples from 30-60 cm and one from 60-90 cm were lost in 2004.

** Three samples from the 60-90 cm layer had SOM content > 400 g kg^{-1}, which is classified as organic soil, and consequently no textural analyses were performed.

depth. The variation in both SOM content and texture was highest at Ribbingsberg, with large variations between plots as well as between layers in the same plot.

When calibrating NIR spectra to SOM and clay content separately, it is important for interpretation of the calibrations to know whether there is any correlation between SOM and clay content. No significant correlations ($P>0.05$, r^2 close to 0) were found between SOM and clay content at Hacksta. At Nybble, however, significant correlations were found in both topsoil and subsoil ($r^2 = 0.31$ and 0.23 respectively). At Ribbingsberg, SOM and clay content correlated significantly ($r^2 = 0.24$) in the topsoil only.

According to the RPD values, the cross-validated estimates of SOM using NIR calibrations would be classified as successful, or even excellent (RPD values between 3.1 and 5.7), except for the subsoil at Hacksta (Table 2). The cross-validated estimates of clay were moderately successful (RPD values between 2.4 and 2.8) except for the topsoil at Hacksta.

Table 2. Correlation between measured and estimated SOM and clay content. Cross-validated NIR-calibrations

		0-30 cm					30-90 cm				
		n	Nr PC	r^2	RMSECV	RPD	n	nr PC	r^2	RMSECV	RPD
Nybble	SOM	52	3	0.93	8.9	4.0	61	4	0.90	9.1	3.1
	Clay	52	3	0.86	38.7	2.8	61	4	0.86	44.5	2.7
Ribbingsberg	SOM	45	5	0.94	13.2	4.2	90	3	0.97	21.8	5.7
	Clay	45	4	0.81	28.5	2.5	90	6	0.86	41.9	2.6
Hacksta	SOM	20	4	0.92	3.0	3.6	40	3	0.66	4.1	1. 7
	Clay	20	2	0.47	57.1	1.4	40	4	0.83	53.6	2.4

n = number of plots without nitrogen fertilisation included in the model
nr PC = number of PLS-components used in the calibration models.
RMSECV = root mean squared error of cross-validation in g kg^{-1}.
RPD = standard deviation divided by RMSECV.

Plant N uptake

Ribbingsberg had on average the highest plant N uptake and often also the largest within-year variation (Table 3). The lowest variation was found at Hacksta. Nybble had the lowest average N uptake and both the uptake and the within-field variation were almost the same in all three years. At Ribbingsberg, the two first years had a similar plant N uptake whereas the third year had both lower and less varied uptake.

Table 3. Nitrogen (kg N ha^{-1}) in aboveground plant parts from 1 m^2 (4 x 0.25 m^2 quadrants) in (*n*) plots without nitrogen fertilisation (= plant N uptake)

	Year	Crop	n	Mean	Min	Median	Max	StDev
Nybble	2003	Oats	20	53	20	45	134	30
	2004	Oats	20	51	21	39	106	29
	2005	Spring barley	12	53	18	55	111	31
Ribbingsberg	2003	Winter wheat	20	91	41	73	167	41
	2004	Oats	13	94	37	95	150	37
	2005	Winter wheat	12	68	32	64	122	26
Hacksta	2004	Winter wheat	21	67	33	70	105	19

When predicting plant N uptake within fields, using NIR or a combination of SOM and clay independently, there were no significant differences ($P>0.05$) between the models based on the topsoil only or those that included data from the whole profile. Table 4 shows statistics for cross-validated calibration models for predicting plant N uptake based on topsoil i) NIR-spectra; ii) SOM and clay content; and iii) a combination of NIR, SOM and clay content. Including the sand and silt fractions did not improve the calibrations (data not shown), hence only SOM and clay content were used. There were no significant differences between the performances of the three different methods or between using absolute or normalised plant N uptake in the calibrations that included data from all three years. The cross-validated estimations of plant N uptake at Nybble were moderately successful (average RPD values of 2.3). This was also the case at Ribbingsberg for the years 2004 and 2005 (average RPD values of 2.1). However, the calibrations for 2003 did not perform as well. The most likely explanation for this was the unintended application of pig slurry in the autumn of 2002 before the experiment started, which presumably masked the variation in soil mineralisation. The calibration models including all three years at Ribbingsberg performed somewhat better than the calibration for 2003, but the poor correlation between plant N uptake and all predictors in 2003 was clearly reflected in the results (Table 4) and 2003 was excluded from further calibrations. At Hacksta, none of the three calibration models tested could accurately estimate plant N uptake.

At Nybble, predictions between years performed almost as well as the cross-validated calibrations for individual years (Table 5, Fig 2a). However at Ribbingsberg, the predictions between the two years (2004 and 2005) gave poor results. There were no significant differences between predictions using NIR spectra or SOM and clay content at Nybble and

Table 4. Correlation between measured and estimated plant N uptake through cross-validated calibration models (0-30 cm) using i) NIR-spectra (PLS-model), ii) SOM and clay content (MLR-model) or iii) NIR-spectra, SOM and clay content (PLS-model). Calibrations for separate years and calibrations including all three years together (absolute and normalised plant N uptake) are shown.

		NIR				SOM and clay			NIR + SOM and clay			
	n	nr PC	r^2	RMSECV	RPD	r^2	RMSECV	RPD	nr PC	r^2	RMSECV	RPD
Nybble												
2003	20	3	0.85	11	2.6	0.69	16	1.8	2	0.87	11	2.9
2004	20	3	0.75	14	2.1	0.81	12	2.3	1	0.81	12	2.3
2005	12	2	0.83	12	2.4	0.79	14	2.3	1	0.64	18	1.7
2003-2005	52	1	0.79	13	2.2	0.77	14	2.1	1	0.81	13	2.3
2003-2005 norm.[*]	52	1	0.78	0.46	2.2	0.76	0.48	2.1	1	0.80	0.43	2.3
Ribbingsberg												
2003	20	2	0.26	36	1.1	0.36	33	1.3	1	0.35	33	1.3
2004	13	2	0.81	16	2.4	0.77	17	2.2	2	0.81	16	2.3
2005	12	6	0.79	12	2.3	0.54	18	1.5	2	0.72	13	1.9
2003-2005	45	1	0.44	28	1.3	0.52	26	1.5	1	0.55	25	1.5
2003-2005 norm.	45	1	0.49	0.69	1.5	0.63	0.59	1.7	1	0.61	0.60	1.7
2004-2005	25	7	0.81	15	2.3	0.64	20	1.7	1	0.63	20	1.7
2004-2005 norm.	25	2	0.81	0.42	2.4	0.74	0.49	2.1	2	0.88	0.34	2.9
Hacksta												
2004	20	6	0.16	17	1.1	0	-	-	-	0	-	-

n = number of plots without nitrogen fertilisation included in the model
nr PC = number of PLS components used in the calibration models.
RMSECV = root mean squared error of cross-validation in g kg^{-1}.
RPD = Standard deviation divided by RMSECV.

[*] A calibration model based on three years with normalised plant N uptake.

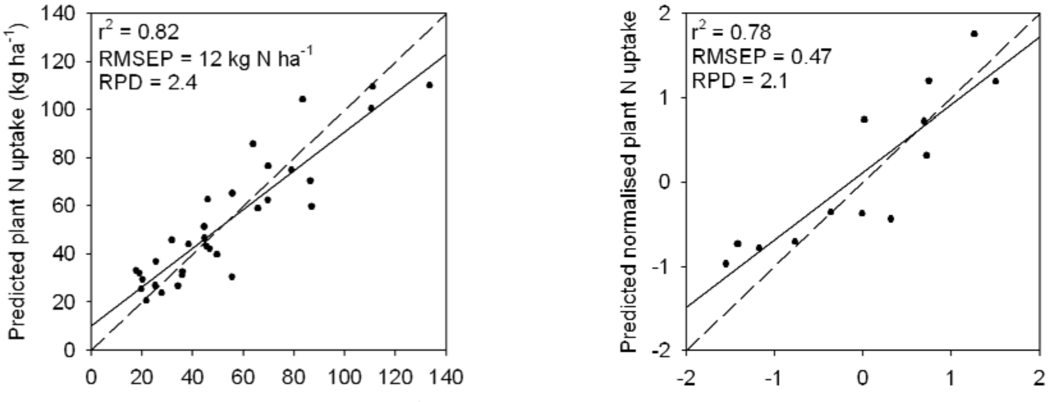
there were no significant differences between using absolute or normalised plant N uptake. Since there were only two predictions between years at Ribbingsberg, no statistically reliable differences could be calculated. However there was a tendency for better predictions using normalised plant N uptake at Ribbingsberg (Table 5, Fig 2b). Because of the lack of data in the subsoil in one year, there were insufficient data for reliable statistical differences between predictions using the whole soil profile or the topsoil only at Nybble. However, the few results from predictions with data from the whole profile did not indicate improved results compared with the predictions from the topsoil (data not shown).

All predictions between fields gave poor results. Predicting plant N uptake at Ribbingsberg using NIR calibration models from Nybble resulted in an average r^2 value of 0.65 and RPD value of 1.0. Predicting plant N uptake at Nybble using NIR calibration models from

Table 5. Predictions of plant N uptake and normalised plant N uptake between years within the same field at Nybble and Ribbingsberg. The calibration models used for the predictions are based on NIR spectra (0-30 cm) or SOM and clay content (0-30 cm).Calibration models including the year 2003 were excluded at Ribbingsberg because of application of farmyard manure.

Years used	Years	NIR					SOM and clay				
for calibration	predicted	r^2	RMSEP	RPD	r^2_{norm}	RPD_{norm}	r^2	RMSEP	RPD	r^2_{norm}	RPD_{norm}
Nybble											
2003	2004+2005	0.82	16	1.8	0.80	1.8	0.80	15	1.9	0.77	1.9
2004	2003+2005	0.82	12	2.4	0.82	2.3	0.77	15	2.0	0.77	1.8
2005	2003+2004	0.56	19	1.5	0.58	1.5	0.82	19	1.6	0.82	1.5
2003+2004	2005	0.89	15	2.0	0.89	1.8	0.86	18	1.7	0.86	1.5
2003+2005	2004	0.75	14	2.0	0.75	2.0	0.85	12	2.3	0.85	2.1
2004+2005	2003	0.85	17	1.8	0.86	2.0	0.81	14	2.1	0.81	2.2
Mean		*0.78*	*16*	*1.9*	*0.78*	*1.9*	*0.82*	*16*	*1.9*	*0.81*	*1.8*
Ribbingsberg											
2004	2005	0.75	25	1.0	0.75	1.2	0.73	27	1.0	0.73	1.9
2005	2004	0.78	26	1.4	0.78	1.5	0.78	30	1.2	0.78	2.1

RMSEP = root mean squared error of prediction in kg ha^{-1}
RPD = standard deviation divided by RMSEP
$_{norm}$ = normalised plant N uptake



a)

b)

Figure 2. Measured verses predicted a) plant N uptake at Nybble 2003 and 2005 predicted using the calibration model for Nybble in 2004 and b) normalised plant N uptake at Ribbingsberg 2004 predicted using the calibration model for Ribbingsberg 2005.

Ribbingsberg resulted in an average r^2 value of 0.47 and RPD value of 1.1 when calibration models from single years were used. The calibration model that included both 2004 and 2005 at Ribbingsberg failed to predict plant N uptake at Nybble. Predicting plant N uptake at Ribbingsberg using calibration models based on SOM and clay content at Nybble resulted in an average r^2 value of 0.60 and RPD value of 1.4. The opposite on the other hand, predicting plant N uptake at Nybble using calibration models based on SOM and clay content from Ribbingsberg failed to give any predictions, with r^2 values close to 0.

Figure 3 shows the scores plot for the first two principal components (PCs) from a principal component analyses (PCA) on NIR spectra from the topsoil. The soil samples are from all the zero N-plots in all three fields. Even though not clearly separated, Ribbingsberg deviates from the other two in the first PC while Nybble and Hacksta tend to separate in the second PC.

Discussion

The predictions of plant N uptake in this study support the suggestion by Börjesson et al. (1999) that the additional information in NIR spectra compared with SOM content largely is information on soil texture. Predicting plant N uptake using NIR spectra or SOM and clay content gave equally good results. Furthermore, no improvements were found when the two methods were combined. This indicates that the spectral features of SOM and clay content are the overall important information in the NIR spectra relating to plant N uptake. In relation to NIR both SOM and clay content are considered primary properties since NIR spectra are directly influenced by combinations and overtones of fundamental vibrations for organic functional groups and water, particle size and surface properties (Chang et al. 2001; Ben-Dore and Banin 1995a). Other soil properties also influencing plant N uptake may in some situations be predicted with NIR spectroscopy due to correlations to more spectrally active properties, for example plant nutrients (Ben-Dore and Banin 1995b), biochemical properties and extractable and exchangeable cat ions (Chang et al. 2001).

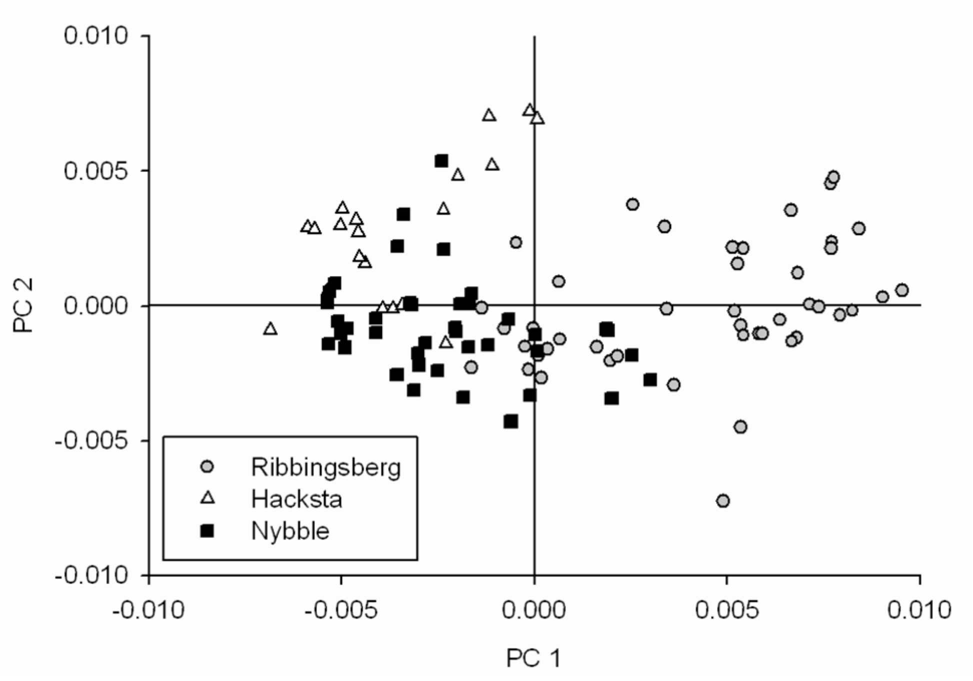


Figure 3. PCA scores plot (the first two principal components) of the NIR spectra from the 0-30 cm soil depth including all three years at Ribbingsberg and Nybble and the single year at Hacksta.

That information on soil texture constitutes a large part of the additional predictive capacity of NIR spectroscopy for plant N uptake compared to SOM content is further supported by the poor results at Hacksta, where the variation in SOM and clay content failed to explain plant N uptake and the same poor results were found for the NIR-based calibrations. This implies that NIR predictions could be limited to fields with a clear relationship between plant N uptake on one side and SOM and texture at the other, consequently fields with quite large variations in SOM content. However, because of the limited number of fields in this study, it can not be said for certain that this always holds true.

Moreover, NIR calibrations for SOM and clay were in general good showing that the information is there. However, if highly correlated, there are reasons to expect that when using NIR spectroscopy to predict the two properties, the same spectral features are used in both calibrations and the less spectrally active property is predicted based on the correlation to the more dominant property. In the present study, there were correlations between SOM and clay content at Nybble and in the topsoil at Ribbingsberg. However, these correlations were weak and considerably weaker than the cross-validated r^2 for predicting SOM and clay, suggesting that they were calibrated independently.

The NIR calibrations for SOM content resulted in high RPD values (~3-4) for all three fields and all soil layers with the exception of the subsoil at Hacksta. The range of SOM in the subsoil at Hacksta was similar to that in the topsoil, but the overall content was lower, <1 to 33 g kg^{-1} compared with 20 to 56 g kg^{-1}, and with half the samples having a SOM content below 1 g kg^{-1}. This, together with the small number of samples (n= 20), possibly explains the poor results. Results from 35 Canadian soils with a similar range in SOM content as the topsoil at Nybble and Ribbingsberg showed standard error of prediction (SEP) values corresponding to 10 g kg^{-1} SOM, r^2 of 0.9 and RPD values between 2.7 and 3.3 (Malley et al. 2000), which are well in line with the results found in the present study. The low RMSEP in the topsoil at Hacksta compared with Nybble and Ribbingsberg (3.0 g kg^{-1} compared with 8.9 and 13.2 g kg^{-1}) can be explained by the smaller range at Hacksta, with the r^2 value remaining above 0.9.

The predictions for clay were also good and in line with earlier studies (r^2 of 0.81-0.86 and RMSECV of 29-39 g kg^{-1}, Table 2). Regional Swedish calibration models have predicted the clay content of independent validation samples with an r^2 of 0.94 and an RMSEP of 39 g kg^{-1} (Stenberg et al. 2002). Sørensen and Dalsgaard (2005) reported r^2 values of 0.95 and RMSECV between 33 and 47 g kg^{-1} for a broad range of Danish soils. Data from the same farm at Hacksta with an NIR calibration for clay content that included 25 samples from the 0-20 cm layer increased the r^2 to 0.88 and reduced the RMSEP to 30 g kg^{-1} (Wetterlind et al. 2007a), indicating that 20 samples are at the lower limit even for such a limited area as 15 ha. Shepherd and Walsh (2002) detected a rapid decrease in predictability when the calibration samples from a large geographical area in eastern and southern Africa decreased below about 100 to 200 samples. However, NIR calibrations with 20 soil samples from another Swedish farm predicted clay content with $r^2 = 0.97$ and RMSEP of 18 g kg^{-1} (Wetterlind et al. 2007b). Smaller calibration sets can presumably perform very well for restricted geographical areas, as interpolation steps are small compared with what can be expected over larger areas.

The results of the NIR calibrations for plant N uptake within fields and years at Nybble and Ribbingsberg (except for 2003 at Ribbingsberg, where the influence of farmyard manure applied in autumn 2002 affected the result), with r^2 values between 0.75 and 0.85 and RPD values over 2.0, are comparable with those obtained in earlier studies (Börjesson et al. 1999;

Stenberg et al. 2005). According to the classification suggested by Mally et al (2004) these results would be moderately useful. However, due to the very complex nature of the soil system Chang et al. (2001) proposed a some what more forgiving classification with good predictions when RPD values exceed 2.0. Considering all the complex functions influencing plant N uptake, including sampling errors and unpredictable parameters such as weather, RPD values over 2 may be a good point of reference for this variable. For implementation in practise, the performance of the method compared to what is done today is also of interest, and an RPD of 2 implies that fertilization errors cold be halved compared to an equal rate all over the field. Dunn et al. (2000) obtained comparable results in one out of two experiments in rice grown in south-eastern Australia, but the other resulted in r^2 values of 0.5, which is in line with results from an earlier study in N fertiliser response experiments in maize throughout Pennsylvania (Fox et al. 1993). However NIR calibrations failed to predict plant N uptake in a rice crop in California (van Groenigen et al. 2003).

Variations in weather and climate conditions are factors strongly influencing plant N uptake. In this study, the effect of variability in weather between years was compensated for by normalising plant N uptake. At Nybble, predictions of absolute plant N uptake between years were almost as good as within years, with RPD values of on average 1.9. At Ribbingsberg, however, the results were not as good (RPD of 1.0-1.4). One of the reasons for this could be the difference in plant N uptake in 2004 and 2005 at Ribbingsberg compared with the very similar plant N uptake in all three years at Nybble. This is supported by the much improved results obtained using normalised N uptake at Ribbingsberg in the calibrations using SOM and clay content (Table 5). However, the NIR calibrations for normalised plant N uptake did not perform as well.

Stenberg et al. (2005) presented promising results for predictions of plant N uptake between fields. This was not achieved in the present study. However the two fields in their study were from the same area and could be expected to be more similar in terms of both soil type and climate compared with the fields in this study. The poor results in predicting plant N uptake between fields show that the relationships between NIR and the soil systems were too different between the fields as indicated in the PCA (Fig 3). This could indicate that calibrating models for large geographical areas with resolution for within-field variations could be problematic, whereas calibrations on field or farm scale, with a more homogeneous soil type, may provide reasonable and useful results. However, since only three fields, one with limited data, were included in this study, further investigations are needed before the possibility of calibration models over larger areas are ruled out.

In addition, one problem with the PLS regression approach used in this study is the possibility of non linearity due to the dynamic and complex nature of the plant N uptake. The PLS regression model can to some extent compensate for nonlinearity by using more components, but only with large calibration data sets which was not the case in this study. With a large database more complex and non linear calibration models could be applied, for example neural networks. However, the limited number of data available in this study would possibly only lead to over fitting (Borggaard 2001).

Another possible development could be to integrate NIR predictions with crop growth models, including both soil and climate, to further improve the predictions.

The results indicate that data from the topsoil are sufficient for predicting plant N uptake and that there is no need for samples from the subsoil. This contradicts the results of Börjesson et al. (1999), who improved predictions of N uptake by including the 30-50 cm layer with the top 20 cm of soil.

Conclusions

The present study supports results from earlier studies on the possibility of improving N uptake prediction accuracy using NIR spectroscopy in fields with large variations in SOM (over 150 g kg^{-1}) compared to using SOM only as predictor. The study also suggests that the additional predictive capacity of NIR compared with SOM at the sites reported here is related to variations in soil texture. The possibility to build prediction models for plant N-uptake for larger geographical areas than within fields needs further investigations, including more and divers fields and taking into account possible nonlinearity in the response.

Acknowledgements

We wish to thank the Swedish Farmers' Foundation for Agricultural Research (SLF) for funding the work and the farmers Sören Nyman, Mikael Fransén and Jarl Ryberg for putting their fields at our disposal. We also wish to thank Thord Karlsson for valuable collaboration during the planning and execution of the field work.

References

Alexandersson H and Eggertsson Karlström C L-M, 2001 Temperaturen och nederbörden i Sverige 1961-1990. Referensnormaler - utgåva 2. pp 71. SMHI, Norrköping.

Barnes RJ, Dhanoa MS and Lister SJ 1989 Standard normal variate transformation and de-trending of near-infrared diffuse reflectance spectra. Applied Spectroscopy 43, 772-777.

Bendor E and Banin A 1995a Near-infrared analysis as a rapid method to simultaneously evaluate several soil properties. Soil Sci. Soc. Am. J. 59, 364-372.

Bendor E and Banin A 1995b Near-Infrared Analysis (Nira) as a Method to Simultaneously Evaluate Spectral Featureless Constituents in Soils. Soil Sci. 159, 259-270.

Borggaard, C 2001 Neural Networks in Near-Infrared Spectroscopy. *In* Near-Infrared Technology in the Agricultural and Food Industries. 2st ed. Ed P Williams and K Norris. St. Paul, MN, Am. Assoc. Cereal Chem.: 101-107.

Börjesson T, Stenberg B, Linden B and Jonsson A 1999 NIR spectroscopy, mineral nitrogen analysis and soil incubations for the prediction of crop uptake of nitrogen during the growing season. Plant Soil 214, 75-83.

Chang CW, Laird DA, Mausbach MJ and Hurburgh CR 2001 Near-infrared reflectance spectroscopy-principal components regression analyses of soil properties. Soil Sci. Soc. Am. J. 65, 480-490.

Cozzolino D and Moron A 2006 Potential of near-infrared reflectance spectroscopy and chemometrics to predict soil organic carbon fractions. Soil Tillage Res. 85, 78-85.

Delin S and Lindén B 2002 Relations between net nitrogen mineralization and soil characteristics within an arable field. Acta Agric. Scand. Sect. B-Soil Plant Sci. 52, 78-85.

Dunn B, Beecher G, Batten G and Blakeney A 2000 Estimating the uptake of nitrogen by rice crops using near infrared reflectance analysis of soil. *In* Near Infrared Spectroscopy: Proceedings of the 9th International Conference, Verona, Italy, 2000. Eds A M C Davies and R Giangiacomo. pp 565-568.

Ekström G 1927 Klassifikation av Svenska Åkerjordar (Classification of Swedish Arable Soils). Sveriges Geologiska Undersökning, Ser C. No. 345 (Årsbok 20), 161 pp.

Fox RH, Shenk JS, Piekielek WP, Westerhaus MO, Toth JD and Macneal KE 1993 Comparison of near-infrared spectroscopy and other soil nitrogen availability quick tests for corn. Agronomy Journal 85, 1049-1053.

Fystro G 2002 The prediction of C and N content and their potential mineralisation in heterogeneous soil samples using Vis-NIR spectroscopy and comparative methods. Plant Soil 246, 139-149.

Gee GW and Bauder JW 1986 Particle-size analysis. *In* Physical and Mineralogical Methods, Ed A Klute. pp 383-411. Soil Science Society of America, Madison.

Hartmann H P and Appel T 2006 Calibration of near infrared spectra for measuring decomposing cellulose and green manure in soils. Soil Biol. Biochem. 38, 887-897.

Islam K, Singh B and McBratney A 2003 Simultaneous estimation of several soil properties by ultra-violet, visible, and near-infrared reflectance spectroscopy. Australian Journal of Soil Research 41, 1101-1114.

Malley DF, Martin PD and Ben-Dor E 2004 Applications in analysis of soils. *In* Near-Infrared Spectroscopy in Agriculture, Eds CA Roberts, J Workman Jr and JB Reeves III. pp 729-784. ASA, CSSA, SSSA, Madison, Wisconsin, USA.

Malley DF, Martin PD, McClintock LM, Yesmin L, Eilers RG and Haluschak P 2000 Feasibility of analysing archived Canadian prairie agricultural soils by near infrared reflectance spectroscopy. *In* Near Infrared Spectroscopy: Proceedings of the 9th International Conference, Eds A M C Davies and R Giangiacomo. pp 579-585. NIR Publications, Chichester, UK.

Martens H and Naes T 1989 Multivariate Calibration. John Wiley & Sons, Chichester, UK. 419 pp.

Mouazen AM, De Baerdemaeker J and Ramon H 2005 Towards development of on-line soil moisture content sensor using a fibre-type NIR spectrophotometer. Soil Tillage Res. 80, 171-183.

Reeves JB, Follett RF, McCarty GW and Kimble JM 2006 Can near or mid-infrared diffuse reflectance spectroscopy be used to determine soil carbon pools? Commun. Soil Sci. Plant Anal. 37, 2307-2325.

Savitzky A and Golay M 1964 Smoothing and differentiation of data by simplified least squares procedures. Analytical Chemistry 36, 1627-1639.

Shepherd KD and Walsh MG 2002 Development of reflectance spectral libraries for characterization of soil properties. Soil Sci. Soc. Amer. J. 66, 988-998.

Sorensen L K and Dalsgaard S 2005 Determination of clay and other soil properties by near infrared spectroscopy. Soil Sci. Soc. Am. J. 69, 159-167.

Stenberg B, Jonsson A and Borjesson T 2005 Use of near infrared reflectance spectroscopy to predict nitrogen uptake by winter wheat within fields with high variability in organic matter. Plant Soil 269, 251-258.

Stenberg B, Jonsson A and Börjesson T 2002 Near infrared technology for soil analysis with implications for precision agriculture. *In* Near Infrared Spectroscopy: Proceedings of the 10th International Conference, Kyongju S. Korea, 2002. Eds A Davies and R Cho. pp 279-284.

Stenberg B, Nordkvist E and Salomonsson L 1995 Use of near infrared reflectance spectra of soils for objective selection of samples. Soil Sci. 159, 109-114.

Terhoeven-Urselmans T, Michel K, Helfrich M, Flessa H and Ludwig B 2006 Near-infrared spectroscopy can predict the composition of organic matter in soil and litter. J. Plant Nutr. Soil Sci.-Z. Pflanzenernahr. Bodenkd. 169, 168-174.

van Groenigen JW, Mutters CS, Horwath WR and van Kessel C 2003 NIR and DRIFT-MIR spectrometry of soils for predicting soil and crop parameters in a flooded field. Plant Soil 250, 155-165.

Wetterlind J, Stenberg B and Söderström M 2007a Farm-soil mapping using NIR-technique for increased sample point density. *In* 6th European Conference on Precision Agriculture, Skiathos, Greece, 3-6 June, 2007. Ed JV Stafford. pp 265-270.

Wetterlind J, Stenberg B and Söderström M 2007b New strategy for farm-soil mapping using NIR to increase sample point density. *In* 12th International Conference, Auckland, New Zealand, 2005. Ed RMW Sumner. pp 299-303

Williams, P. C. 1987 Variables Affecting Near-Infrared Reflectance Spectroscopic Analysis. *In* Near-Infrared Technology in the Agricultural and Food Industries. 1st ed. Ed P Williams and K Norris. St. Paul, MN, Am. Assoc. Cereal Chem.: 143-167.

Zadoks JC, Chang TT and Konzak CF 1974 A decimal code for the growth stages of cereals. Weed Research 14, 415-421.