Measurement and analysis of soil nitrogen and organic matter content using near-infrared spectroscopy techniques[*]

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Abstract: Near infrared reflectance (NIR) spectroscopy is as a rapid, convenient and simple nondestructive technique useful for quantifying several soil properties. This method was used to estimate nitrogen (N) and organic matter (OM) content in a soil of Zhejiang Province, Hangzhou County. A total of 125 soil samples were taken from the field. Ninety-five samples spectra were used during the calibration and cross validation stage. Thirty samples spectra were used to predict N and OM concentration. NIR spectra of these samples were correlated using partial least square regression. The regression coefficients between measured and predicted values of N and OM was 0.92 and 0.93, and SEP (standard error of prediction) were 3.28 and 0.06, respectively, which showed that NIR method had potential to accurately predict these constituents in this soil. The results showed that NIR spectroscopy could be a good tool for precision farming application.

Key words: NIR spectroscopy, Partial least square, Precision farming, Soil spatial variability, Nitrogen, Organic matter

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INTRODUCTION

Technological development in positioning, sensing and control system has opened a new era, in which traditional agricultural practices are left behind. Precision farming is a term used to describe the management of variability within a field, applying agronomic inputs in the right place, at the right time and in the right quantity to improve the economic efficiency and diminish the adverse environmental impact of crop production (Earl *et al*., 2000). Study and understanding of the soil spatial variability is very important as soil variability causes crops yield to be distributed unevenly in a field (Liu *et al*., 1999). The

first step in addressing the problems caused by soil variability is to develop a Site Specific Crop Management program (SSCM) to help improve soil quality through practical and economically efficient treatments. Study of soil constituents' composition involves exhaustive laboratory analysis.

Visible-NIR spectroscopy is a rapid and nondestructive analytical technique that correlates diffusely reflected near-infrared radiation with the chemical and physical properties of materials (Chang and Laird, 2002) and has been used for assessing grain and soil qualities (Morra *et al*., 1991; Ben-Dor and Banin, 1995; Kawamura *et al*., 1999; Delwiche and Hruschka, 2000; Barton *et al*., 2000; Confalonieri *et al*., 2001; McCarty and Reeves III, 2001; Chang and Laird, 2002; Reeves *et al*., 2002; McCarty *et al*., 2003; Kamrunnahar *et al*., 2003) and has been shown to be rapid, convenient, simple, accurate and able to analyze many constituents at the same time. McCarty *et al*.(2003) proposed the spectroscopic techniques for

measuring phosphorus (P), nitrogen (N), and carbon (C) in the environment and for assessing the fate and transport of these nutrients. Considering that organic matter in soil has very complicated chemistry and its strong absorption there can mask some features associated with soil minerals and has profound influence on the physical, chemical and biological properties of soils (Chang and Laird, 2001; Francioso *et al.*, 2000; Shepherd and Walsh, 2002). The goal of this study is to evaluate the potential of NIR spectroscopy to estimate N and OM content in mixed loamy soil, and obtain its distribution maps by using Geographic Information System (GIS).

MATERIALS AND METHODS

Field description

The experimental site located in Hangzhou (120°11′ E, 30°28′ N) Zhejiang, in relatively flat field with monthly temperature fluctuating from 3.5 °C in January to 26.8 °C in July and annual average temperature 16.2 °C and rainfall 279 mm. It has a wet climate, with a long frost-free period and relatively high summer temperatures. The soil normally does not need mechanized tilling, is easy to cultivate by the method of crop rotation with corn. The soil type is classified as loamy mixed active thermic aeric soil according to Zhejiang soil classification.

Sampling strategy and experimental design

The test field boundary was determined with Trimble AgGPS 132 equipment. The normal grid method was used to sample the soil. Measurements were made at 30 sample plots, at a depth of 20 cm and grid interval of 5 m (two sample plots were taken at the diagonal of the south-east grid). A composite sample was obtained by mixing 5 soil samples; 1 central plot and the remaining 4, were separated 1 m away from each sampling point. Other 95 samples were randomly taken from the field to complete the 125 samples to be analyzed by spectroscopy. The collected soil was divided into two portions A and B. A was placed in a properly closed bags and taken to the soil science laboratory and analyzed for N and OM content. Group B was air dried and sieved <2 mm, and then analyzed by NIR spectroscopy.

Soil spectral data measurement and analysis

Soil spectra data collection was done with a spectrophotometer (ASD FieldSpec Pro FR (350~2500 nm)/A110070). The soil was set in a petri dish whose surface was flushed beforehand. Three reflectance spectra were taken over the central area of the petri dish rotating the sample in-between each reflectance spectra approximately by 120°, for each reflectance spectra the scan number was 20 at exactly the same position, a total scan for each sample were 60, fixed scanning time was 0.1 s (Fig.1).
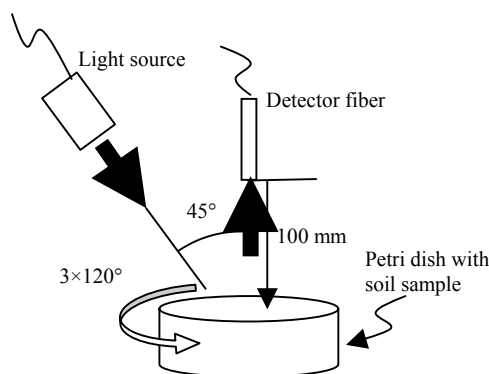

Fig.1 Scheme of the measuremens during spectroscopy data collection

The absorbance of the scanned sample was recorded ($\log(1/R)$). All spectra recorded were checked visually and averaged using ViewSpec pro version 2.14 and sent to multivariate analysis software-the Unscrambler 8.0 (CAMO ASA, Norway). Representative reflectance spectra of some soil samples are shown in Fig.2.
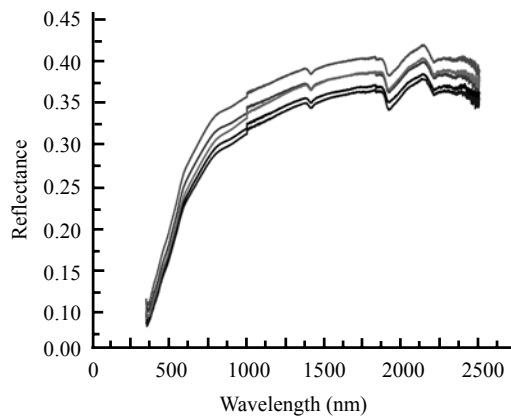

Fig.2 Representative reflectance spectra of some soil samples

NIR spectroscopy is basically an empirical method, whose complete procedure consists of two stages: (set I) calibration stage where the prediction equation is developed and (set II) validation stage where the previous stage is validated. In the calibration stage, 95 soil samples were used. Calibration equations were developed using principal component analysis (PCA) and partial least square (PLS) regression techniques enabling the relating of near-infrared reflectance spectra to measured values of N and OM in the soil. Multiplicative scatter correction (MSC) technique was used as pretreatment to reduce the effect of scattering. Separate calibration equations were computed for each constituent. Leave-one-out cross-validation was done on the calibration set to determine the optimum number of factors (F) for the PLS regression calibration model of each soil property. The F giving the smallest RMSECV (root mean square error of cross-validation) between measured and predicted values was chosen for the PLS regression calibration models. To avoid over-fitting, F was always less than 1/10 of the number of samples in the calibration set. The spectral reflectance data, in units of $\log(1/R)$, were smoothed over 7 (N) and 11 (OM) wavelength points. Each calibration equation developed from set I was used to predict the constituent values for the independent spectra in set II (30 samples); the external prediction set. Results of calibration and cross validation of the best models found using PLS method for N and OM content future prediction are shown in Table 1. The R (correlation coefficient), RMSEP (root mean square error of prediction), and SEP (standard error of prediction) between the predicted and measured values of N and OM were used to evaluate the prediction ability of NIR spectroscopy technique.

RESULTS AND DISCUSSION

Spectral properties and prediction

The reflectance spectra data collected were simi-

lar. All the spectra have three large absorption peaks ($\log(1/R)$) in the near infrared region (700~2500 nm) around 1400, 1900 and 2200 nm (Fig.3), which correspond with the highest absorbance peaks obtained by Chang and Laird (2002) and Fidencio *et al*.(2002); testing soil, and some few small peaks in the region between 1000~1100 nm and 2200~2300 nm.
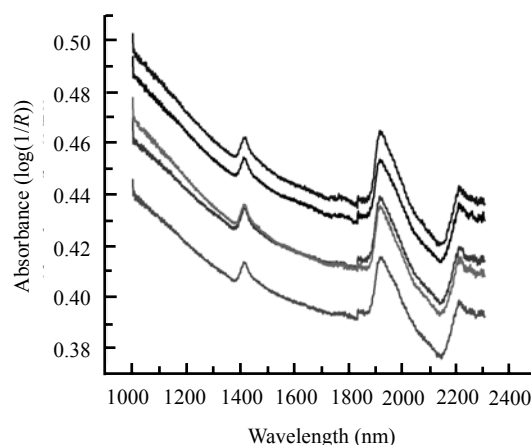

Fig.3 Representative absorbance spectra of some samples in the analyzed region (1000~2300 nm)

Since PLS technique applies linear transform to the entire individual wavelength data, it would be helpful to examine how each soil constituent is simply related to individual wavelength, and the full information was obtained by using the entire individual wavelength so that better understanding of NIR spectra may be obtained. The correlation coefficient across the entire spectral region of 350~2500 nm was in general not high, however, for each constituent during the calibration model construction were found some slightly better coefficients related to individual wavelengths, for example: in N 1902, 2364, 1826 and 2098 nm, only 1826 nm correspond to similar wavelength selected by Morra *et al*.(1991), using multiple linear regression (MLR) technique. The best correlation values for OM were found in 993, 1080, 1951, and 2277 nm, while only 1080 correspond with the wavelength selected by Krishnan *et al*.(1980). In the

Table 1 Results of calibration and cross validation of the best models found using PLS method

Soil constituent	Gap sizes	F	Calibration				Cross-validation			
			R	*SEC*	*RMSEC*	Bias	*R*	*SECV*	*RMSECV*	Bias
N	7	7	0.97	2.05	2.04	1.97e-6	0.94	2.82	2.8	0.02
OM	11	5	0.96	0.07	0.06	−6.79e-8	0.96	0.06	0.06	−0.9e-3

previously selected wavelength not one repeats in both constituents.

The prediction set showing that NIR can predict N and OM in soil accorded with the findings of Ben-Dor and Banin (1995), Fidencio *et al.*(2002), Sudduth and Hummel (1991), Chang and Laird (2002), and Martin and Malley (2003). Statistically for predicted N and OM is shown in Fig.4. The R between measured and predicted values of N and OM being 0.92 and 0.93, as well as SEP 3.28 and 0.06 respectively, showed that NIRS method had potential for predicting accurately these constituents in this soil type. Even though OM percent composition was not above 2%, a slightly better correlation was found in this constituent prediction, although the difference was not quite significant. The *R*=0.93 obtained in the OM prediction was within the range proposed by Martin and Malley (2003) in their investigation on the same constituent (0.81~0.97) and considered as a good approximation. In the case of N, the *R*=0.92 obtained showed also good accuracy, that was lightly higher than that obtained by Morra *et al.*(1991); and Chang and Laird (2002), respectively.

Soil N and OM spatial variability

The obtained (as reference parameters) and predicted, N and OM values were both analyzed on the aspect of their constituents spatial variability. The tested field can be considered as fairly productive based on the N average composition of 70.22 mg/kg ranging from 58.12 to 84.13 mg/kg, and OM average composition of 1.28% ranging from 1.06% to 1.65%.

Predicted and measured (reference) N and OM spatial distributions were interpolated by ArcView GIS 3.1 software using spline method, spatial distribution maps were generated as shown in Fig.5. The reference maps for the predicted and measured (reference) values of N and OM were almost the same. The decreasing westward N concentration was mainly caused by management practices in tillage and irrigation. Quite similar variability was observed in the OM concentration in the field. Through the range of concentration and colors represented in each map, a slight few difference can be found between predicted and reference parameters, which showed that the predicted N and OM through NIR techniques can be used to explain the spatial distributions of this soil. So the NIR as a rapid technique that can be combined with GIS and precision farming.

CONCLUSION

This investigation showed NIRS is a technique that can be considered to have good potential for assessing soil N and OM content. The regression coefficients between measured and predicted values of N and OM were 0.92 and 0.93, and SEP 3.28 and 0.06, respectively.

The spatial distribution map of the soil N and OM showed that NIRS can be useful in situ as a rapid technique that can be combined with GIS and precision farming principles application.
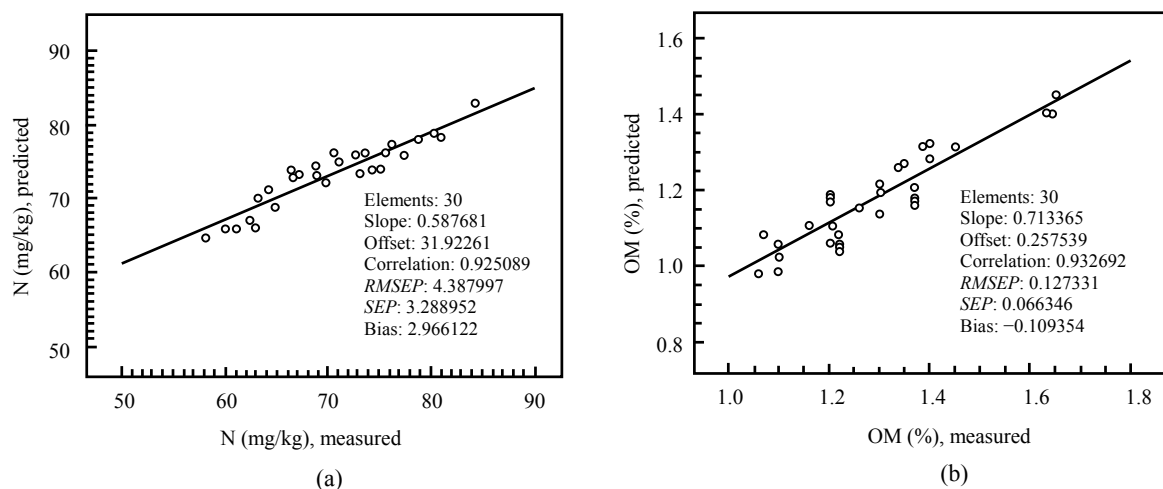

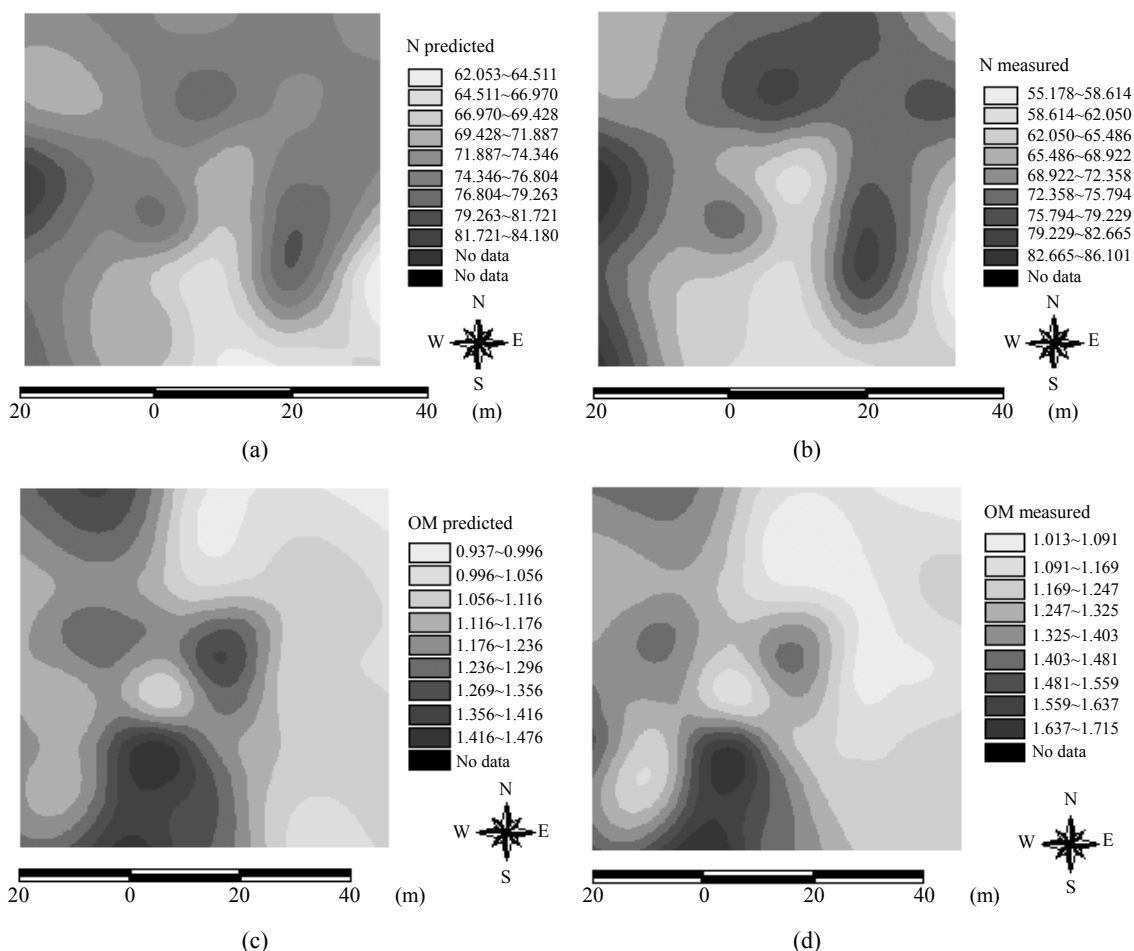**Fig.4 Correlation between measured and predicted values of (a) N and (b) OM**

Fig.5 Spatial distribution maps of predicted and measured (reference) N and OM content (a) N predicted; (b) N measured; (c) OM predicted; (d) OM measured

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