Evaluation of Vertosol soil fertility using ultra-violet, visible and near-infrared reflectance spectroscopy

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Abstract

Precision agriculture needs to match nutrient availability to plant needs so that nutrients will not be a limiting factor. However, nutrient levels are often highly variable at a field scale and the assessment of nutrient levels using traditional soil testing methods is time consuming and expensive. We assessed the capability of ultra-violet (UV), visible (VIS) and near infrared (NIR) reflectance spectroscopy as a rapid and non-destructive analytical method to predict a range of soil fertility parameters in Vertosols from the Liverpool Plains in northern New South Wales. A total of 588 surface and subsurface soil samples (0-150 cm) were used. Partial least square regression (PLS) was used to develop calibration models between reflectance spectral data and measured values of soil properties obtained using traditional laboratory methods. PLS models successfully predicted pH, electrical conductivity (EC), organic carbon, total nitrogen, cation exchange capacity (CEC), exchangeable calcium, magnesium, sodium, Ca:Mg, exchangeable sodium percentage (ESP), manganese, iron, boron and copper (r²=0.46-0.75). In contrast, poor predictions (r²<0.30) were obtained for ammonium and nitrate nitrogen, phosphorus, sulfur, zinc and chlorine. The study demonstrates that reflectance spectroscopy in UV-VIS-NIR range has the potential for rapid prediction of several soil fertility parameters.

Key Words

Soil fertility, precision agriculture, reflectance spectroscopy, soil testing, partial least square regression

Introduction

It is well recognized that soil variability influences the productive potential of agricultural lands. Maps of soil fertility and physical attributes are being used in precision crop management to determine the responsive and non-responsive parts of fields (Wolkowski and Wollenhaupt 1995). Nielsen *et al.* (1995) identified several important soil fertility attributes, including available soil nitrogen, other macro and micro plant nutrients, relative position and slope of the terrain and soil organic matter content that could be mapped and managed for improved yield. Quantification of soil fertility data to explain within-field spatial variation requires intensive collection of soil samples and their subsequent laboratory analysis. At present to obtain the large amount of data through sampling and analysis is time-consuming and expensive. In addition many conventional laboratory methods for analyzing soil properties suffer from analytical problems and inefficiencies such as reproducibility, reliability, time and labour factors (Malley *et al.* 1999). Reflectance spectroscopy techniques provide a possible alternative, to conventional laboratory methods of soil analysis. They require little sample preparation and they are fast, cost-effective, non-destructive, and non-hazardous. Furthermore several constituents can be predicted simultaneously (Batten 1998).

Recent research has confirmed the potential of reflectance spectroscopy in soil based studies (Janik *et al.* 1998). For example diffuse reflectance spectroscopy in ultra-violet (UV, 250-400 nm), visible (VIS, 400-700 nm) and near-infrared (NIR, 700-2500 nm) ranges was successfully used for the rapid characterization of several soil properties, pH, organic carbon, air-dry gravimetric water content, clay, cation exchange capacity (CEC), exchangeable Ca and exchangeable Mg (Islam *et al.* 2003) and some success for sensing soil organic matter in the field (Sudduth and Hummel 1993). In this study we examined the feasibility of using the UV-VIS-NIR diffuse reflectance spectroscopy technique to simultaneously predict several important soil fertility parameters including pH, electrical conductivity (EC), organic carbon, total nitrogen (N), ammonium nitrogen (NH₄-N), nitrate nitrogen (NO₃-N), phosphorus (P), sulphur (S), cation exchange capacity (CEC), exchangeable calcium (Ca), magnesium (Mg), potassium (K) and sodium (Na), calcium and magnesium ratio (Ca:Mg), exchangeable sodium percentage (ESP) and micronutrients including such as manganese (Mg), zinc (Zn), iron (Fe), boron (B), chlorine (Cl) and copper (Cu).

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Study area

The Liverpool Plain located in the northern New South Wales is one of the most fertile and productive agricultural lands in Australia. It is well known for dry land farming including 1500 farms from range to plain. Soil of this area consists of black earths, grey clays and brown clays (Liverpool Plains Catchment: Catchment description 2004)

Materials and methods

Soil sampling

Soil samples were collected from two 100 m transects (25 m apart) taking samples from 60 sites and 5 different depth intervals (0-10 cm, 10-30 cm, 30-60 cm, 60-100 cm, and 120-150 cm) covering Mullaley, Premer, Spring Ridge, Quirindi, Werris Creek and Breeza areas of the Liverpool Plain. Sample sites concentrated on Vertosols used for continuous cropping and were equally divided between flat plains landscapes and gently undulating lower slopes landscapes. Total 558 surface and subsurface soil samples were used in this study. A set of 558 surface and sub-surface soil samples collected from the Liverpool Plain of New South Wales, Australia were used to develop calibration and validation models in the study.

Chemical analysis

Chemical analyses of the soil samples were conducted by Incitec-Pivot Laboratory, Melbourne using standard methods (Table 1).

Method ^a
1:5 soil/water suspension,
1:5 soil/ CaCl ₂ suspension
1:5 soil/water suspension
Texture conversion ^b
Walkley and Black
Kjeldahl
1M KCl extraction
1:5 soil/water suspension
Bicarbonate extraction
$Ca_3(PO_4)_2$ extraction
Sum of exchangeable cations
NH ₄ OAc extraction
Calculation from CEC and exchangeable Na
DTPA extraction
Hot CaCl ₂ extraction
1:5 soil/water suspension

^aRayment and Higinson 1992; ^bSlavich and Peterson 1993

Spectral measurement

The spectral reflectance of soil samples was measured using a Varian Cary 500 UV-VIS-NIR spectrophotometer equipped with a diffuse reflectance accessory (Labsphere DRA-CA-50D) at 1.1 nm intervals in the UV-VIS (250-700 nm) range and 3 nm intervals in the NIR (700-2500 nm) range. For each sample, approximately 20 g of air-dried soil was placed into a sample holder with a quartz window. A standard sample supplied by the instrument manufacturer was used as a reference material for baseline correction. It required approximately three and half minutes to scan a soil sample.

Soil sample selection for calibration and validation sets

To develop the statistical model for each soil property, the total samples (n=588) were randomly divided into two sets. One set was used to develop a prediction equation (calibration set), and the other set was used to validate the predictive equation (the validation set).

Data pretreatment

Soil spectra consisting of 1065 data points within 250-2500 nm range were smoothened by Savitzky and Golay technique (Savitzky and Golay 1964) to spectra of 446 data points (5 nm interval and a filter size of 5). This technique is a simplified least squares procedure for smoothing and differentiating the spectral data, which had been used by researchers to improve spectral quality (Ehsani *et al.* 2001). The distributions of chemical values for most soil properties were positively skewed and to obtain

approximately normal distribution logarithmic transformations were applied prior to multivariate analysis (Atkinson 1985).

Statistical analysis

Partial least square regression (PLS) was used to relate reflectance spectra with measured soil properties. PLS is an example of a latent variable regression method (Martens and Naes 1987), where PLS components known as factors are selected to maximize both description of the independent variables and correlation to the dependent variables. The factors extracted for the data are a function of values of both the independent and the dependent variables; and the two steps of reduction in data dimensionality and regression are performed simultaneously. The calibration equation obtained using PLS can be summarized as follows:

Soil fertility parameter = $C_0 + C_1WV_1 + C_2WV_2 + \dots + C_{446}WV_{446}$ (i)

where C_0 is a constant, $C_1, C_{2, \dots, M}C_{446}$, are regression coefficients and $WV_1, WV_2, \dots, WV_{446}$ are the reflectance values at each wavelength used for the calculations.

The calculations of partial least square regression were performed using the JMP version 5 software (SAS 2002).

Prediction quality

The ability of the UV-VIS-NIR reflectance technique to predict a soil property was evaluated using statistical parameters commonly used for the NIR techniques. One example is the coefficient of determination of measured and predicted values of soil samples (r^2), which measures the proportion of the total variation accounted for by the model. The remaining variation is attributed to random error. The standard error of calibration (SEC) is the standard deviation of the difference between the measured and the estimated values for samples in the calibration set; whereas the standard error of prediction (SEP) is the standard deviation of the estimated values for samples in the validation set. SEC and SEP were calculated from the following equation:

SEC or SEP = $[\Sigma(y-x)^2 / n-1]^{1/2}$ (ii)

where y is the predicted value of a soil property estimated using the UV-VIS-NIR technique, x is the measured value of the same soil property by standard laboratory method and n is the total number of samples.

Another important statistical parameter used to evaluate calibrations was the RPD. The RPD is the ratio of the standard deviation (s.d.) of the measured value of a soil property in the prediction set to the SEP (Starr *et al.* 1981)

RPD = s.d. / SEP (iii)

Outlier detection

Outlier prediction is an important factor during the calibration modelling and monitoring phases (Workman 1992). In this study, an outliers were defined as samples having a difference between measured and predicted values larger than three times SEC or SEP (Chang *et al.* 2001) and subsequently excluded from the calibration and validation models (Islam *et al.* 2003) (Table 3).

Results and discussion

Standard descriptive statistics of the soil properties of the calibration and validation sets reveals a high degree of correspondence (Table 2), which indicates that these data are suitable for modeling. Usually in most reflectance spectroscopy techniques, the best calibration is considered the one with the highest coefficient of determination (r^2), and the lowest standard error of cross validation (SECV). Using cross validations on a data set generally gives an over-optimistic indication of the actual performance of the model. When new and totally independent samples are predicted with a "young" calibration model, it is very rare to get an SEP at the same level as the SECV (Dardenne *et al.* 2000). However in this study we have attempted to develop calibration models using separate validation set of samples (as have Dunn *et*

al. 2002 and Islam *et al.* 2003) expecting similar performance for predicting soil properties of unknown soil samples. We also calculated RPD values (SEP compared with standard deviation) for PLS models to evaluate the performance of the UV-VIS-NIR reflectance technique (Williams and Norris, 1987). Chang *et al.* (2001) classified NIR predicted soil properties into 3 categories based on RPD values in the ranges of >2.0, 1.4-2.0 and <1.4 to indicate decreasing reliability of the prediction models. Considering the above RPD ranges, we were able to successfully predict several soil fertility parameters of Liverpool Plain Vertosols in this study. The results are presented in Table 3.

Soil properties	Calibration set					Validation set				
	n	Mean	Min.	Max.	s.d.	n	Mean	Min.	Max.	s.d.
pH (1:5 H ₂ O)	293	8.6	6.6	10.0	0.7	293	8.6	6.7	10.0	0.6
pH (1:5 CaCl ₂)	287	7.9	6.1	9.6	0.6	284	7.9	6.1	9.4	0.6
EC (1:5 H ₂ O) (mS/cm)	277	0.3	0.1	1.2	0.2	277	0.3	< 0.1	1.3	0.2
EC (SE) (mS/cm)	274	2.1	0.5	8.9	1.2	271	2.0	0.4	8.9	2.0
OC (%)	291	0.9	0.1	2.1	0.4	286	0.8	0.1	2.1	0.4
Total N (g/kg)	290	0.6	0.1	2.0	0.3	290	0.6	0.1	2.0	0.3
NH ₄ -N (mg/kg)	294	4.8	0.9	91.0	7.3	294	5.2	0.9	102.0	9.4
NO ₃ -N (mg/kg)	271	9.1	0.2	65.0	9.0	260	8.3	0.2	42.0	7.6
P (mg/kg)	288	32.5	1.0	120.0	25.1	286	31.9	1.0	130.0	24.2
S (mg/kg)	294	38.8	1.4	2519.0	176.0	294	42.1	1.4	3239	222.0
CEC (mmol _c /kg)	291	688.4	411.0	924.0	93.9	289	686.1	393.0	869.0	95.9
Exch. Ca (mmol _c kg)	292	313.4	90.1	499.3	80.1	293	313.2	85.2	508.7	81.3
Exch. Mg (mmol _c /kg)	292	313.2	79.0	526.5	89.4	293	312.2	82.0	544.1	89.6
Exch. K (mmol _c /kg)	288	12.6	2.3	29.0	4.5	289	12.6	2.7	27.5	4.5
Exch. Na (mmol _c /kg)	273	38.0	1.5	290.0	43.4	257	33.3	1.2	271.6	39.8
Ca:Mg	289	1.1	0.2	4.1	0.7	292	1.2	4.1	0.2	0.7
ESP %	276	5.4	0.2	41.5	6.3	278	5.5	0.2	37.4	6.3
Mn (mg/kg)	257	6.4	0.8	25.0	3.7	265	6.9	0.9	32.0	4.7
Zn (mg/kg)	294	1.2	0.2	15.9	1.6	294	1.3	0.2	10.4	1.6
Fe (mg/kg)	289	31.9	9.0	71.0	10.7	288	31.8	0.9	64.0	10.5
B (mg/kg)	277	1.9	0.2	7.4	1.3	275	1.7	0.6	5.4	0.9
Cu (mg/kg)	289	1.9	0.4	3.4	0.5	290	1.9	0.4	3.5	0.5
Cl (mg/kg)	294	82.0	4.0	2050.0	245.0	294	81.3	4.0	1900.0	238.0

Table 2. Statistical summary of soil properties for the soil samples used in the calibration and validation models of the study.

n, number of samples; s.d., standard deviation

Plant nutrients such as N, P, S, Mn, Zn, Fe, B, Cu, and Cl exists in soil as organic complexes, chelates, salts and in ionic forms. Theoretically, NIR responds to rotational and vibrational bonding energies of hydrogen, therefore inorganic salts would be transparent to the NIR energy (Clark *et al.* 1987). However NIR may determine some cation concentrations depending on their association with organic and hydrated inorganic molecules (Clark *et al.* 1987). In this study, the calibration model developed for organic carbon might be said to be a direct measurement as various functional groups of organic matter (-CH, -OH, -NH) absorbs NIR radiation (Williams and Norris 1987). Measurement of soil fertility parameter including macro and micro-nutrients would be due to their inter-correlations with organic matter and /or clay and might be said to be indirect measurements.

In this study, predictions of pH were compared between two commonly used laboratory methods. Prediction accuracy was found to be slightly better for pH measured in water extracts (r^2 =0.62, SEP=0.39 and RPD=1.6) than pH measured in CaCl₂ extracts (r^2 =0.48, SEP=0.41 and RPD=1.4). Soil samples in this study were mostly alkaline (pH (1:5 H₂O) mean=8.6, range=6.6-10.0). Islam *et al.* (2003) reported a higher level of accuracy (r^2 =0.71, SEP=0.61 and RPD=1.8) for a calibration model developed on soil samples with a wider range of pH values (pH (1:5 H₂O), mean=7.2, range=3.7-9.7).

Soil properties	Calibration set				Validation set				
	n	outliers	r^2	SEC	n	outliers	r^2	SEP	RPD
pH (1:5 H ₂ O)	293	1	0.68	0.37	293	1	0.62	0.39	1.6
pH (1:5 CaCl ₂)	287	7	0.44	0.43	284	10	0.48	0.41	1.4
EC* (1:5 H ₂ O) (mS/cm)	277	17	0.75	0.09	277	17	0.71	0.10	1.8
EC* (Sat. extract) (mS/cm)	274	20	0.51	0.84	271	23	0.46	0.80	1.4
OC (%)	291	3	0.75	0.20	286	8	0.70	0.22	1.8
Total N* (g/kg)	290	5	0.61	0.19	290	4	0.50	0.22	1.4
CEC /mmol ₊ kg ⁻¹	291	3	0.76	45.8	289	5	0.75	48.8	2.0
Exch. cations /mmol ₊ kg ⁻¹									
Ca	292	2	0.62	49.9	293	1	0.50	58.4	1.4
Mg	292	2	0.70	50.0	293	1	0.61	56.2	1.6
Κ	285	9	0.42	3.4	289	7	0.33	3.8	1.2
Na*	273	21	0.55	28.5	268	29	0.57	26.3	1.5
Ca:Mg*	287	7	0.70	0.40	287	7	0.64	0.37	1.8
ESP* %	276	18	0.64	3.8	278	16	0.58	4.2	1.5
Mn* (mg/kg)	257	37	0.73	2.0	265	29	0.73	2.4	1.9
Fe (mg/kg)	289	5	0.68	0.04	288	6	0.60	6.8	1.6
B* (mg/kg)	277	17	0.61	0.82	257	37	0.59	0.61	1.5
Cu* (mg/kg)	289	5	0.47	0.35	290	4	0.47	0.38	1.4

Table 3. Predictions within calibration and validation sets using the partial least square regression (PLS) model.

n, number of samples; r^2 , coefficient of determination for measured and predicted values; SEC, standard error of calibration; SEP, standard error of performance; RPD, ratio of standard deviation of validation set to SEP; * logarithmic transformations were applied to chemical values

The EC calibration developed on 1:5 soil/water suspensions was more accurate ($r^2=0.71$, SEP=0.10 and RPD=1.8) than the EC converted to saturation extract using a texture conversion factor ($r^2=0.46$, SEP=0.80 and RPD=1.4). We have predicted organic carbon with a RPD value of 2.0 ($r^2=0.70$, SEP=0.22), which was better than the predictive models reported by other researchers (McCarty *et al.* 2002; Dunn *et al.* 2002).

Total N was determined with a RPD value of 1.4 ($r^2=0.50$) where as r^2 values of 0.85 (Chang *et al.* 2001) and 0.96 (Reeves *et al.* 1999) have been reported by others. We were unable to predict NH₄-N ($r^2<0.1$ and RPD<1.0), NO₃-N ($r^2=0.42$ and RPD=1.3), P (Colwell) ($r^2=0.38$ and RPD=1.2) and S ($r^2<0.1$ and RPD<1.0) (data not presented). The reason could be the narrow ranges of these soil properties or poor correlations between these nutrients and organic carbon.

CEC ($r^2=0.75$, SEP=4.8 and RPD=2.0), exchangeable Ca ($r^2=0.50$, SEP=58.4 and RPD=1.4), Mg ($r^2=0.61$, SEP=56.2 and RPD=1.6), Na ($r^2=0.57$, SEP=26.3 and RPD=1.5), Ca:Mg ($r^2=0.64$, SEP=0.37 and RPD=1.8) and ESP (($r^2=0.58$, SEP=4.2 and RPD=1.5) were well predicted (Table 3) and similar results were reported by Dunn *et al.* (2002) while investigating the Riverine Plain Soils from eastern Australia. The poor prediction accuracy for exchangeable K ($r^2=0.33$, SEP=3.8 and RPD=1.2) might be due to its narrow range within the data set (range=2.3-29.0 mmol_c/kg).

Among micronutrients we were able to predict Mn (RPD=1.9), Fe (RPD=1.6), B (RPD=1.5) and Cu (RPD=1.4) but unable to predict Zn and Cl, which might be again due to narrow ranges of these nutrients within the data set. Figure 1 represents validation plots of some soil fertility parameters.



Figure 1. Plots of the predicted vs. measured values for some soil fertility parameters (n, number of soil samples in the validation stage; r², coefficient of determination for measured and predicted values and SEP, standard error of prediction; RPD, ratio of standard deviation of validation set to the standard error of prediction.

Conclusions

Predicting nutrient elements and other soil fertility parameters such as CEC and exchangeable bases is challenging as there is no direct relationship between reflectance in the UV-VIS-NIR ranges and these soil parameters. Calibration might be possible depending on the degree of correlation between organic matter and/or clay and soil fertility parameters. Nevertheless, we were able to predict CEC at a reasonable level. Also, we were able to predict pH, EC, organic carbon, total N, exchangeable Ca, Mg, Na, Ca:Mg ratio, ESP and micro-nutrients including as Mn, Fe, B and Cu with an acceptable level of accuracy (r^2 =0.46- 0.75) and prediction was poor for exchangeable K, NH₄-N, NO₃-N, P, S, Zn and Cl (r^2 <0.30). The poor predictability of these fertility parameters may be due to the narrow range of their values in the data set.

To be practically used for site-specific crop management a higher level of prediction accuracy (r^2 >0.80) is required for the above soil parameters, where as our validated r^2 ranges from 0.46-0.75. But given the relative speed and cost of this technique compared to traditional laboratory methods; and large local variations of soil properties, we suggest that the ability to analyse a large number of samples at finer sampling intervals using the spectroscopic technique will outweigh the deficiency in analytical precision. Further research for understanding the nature of the relationship between soil reflectance spectra and soil nutrients may be needed by taking samples representing different soil types, land use and climatic conditions.

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