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Review Article

Near Infrared Spectroscopy based Soil Nitrogen measurement - A Review

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Abstract

Spectroscopy is a rapid, simple, non-destructive and analytical technique, which has been increasingly used for agricultural and food analysis processes. Soil nitrogen, being an important macronutrient of the soil, measurement of total Nitrogen (TN) content can be used as an index of crop productivity. Near infra-red (NIR) spectroscopy can accurately predict the soil nitrogen content by evaluating reflected Near Infra-red rays intensity. The need of expensive and bulky spectrometers is eliminated by use of light-emitting diode (LED) as a Near Infra-red light source, making the system cost effective and portable. The paper reviews brief study of different technologies used for the purpose and explains in detail the spectroscopy concept. A comparative study between various modern calibration methods has lead to a conclusion that, wavelet analysis can offer utmost accuracy of prediction.

Keywords: *Spectroscopy, NIR, Total Nitrogen, Spectrometers, LED, Artificial Neural Networks, wavelet.*

1. Introduction

Over the years, soil erosion and continuous cropping without adequate nutrients inputs, result to soil health deficiency in rain-fed areas. To increase crop productivity, the balanced nutrient management approach should be introduced to farmers. Soil samples provide a representation of the ability of the soil to supply nutrients to meet crop needs throughout the growing season, as well as support the desired yield. To increase crop productivity, we must know what nutrients are present in the soil and in what amount. A precise soil analysis can only result in more efficient fertilizer use, increased yield and low expenditure.

Amongst the various nutrients obtained from soil, six nutrients are utilized in large amounts and hence are called as macronutrients. From these, nitrogen, phosphorus and potassium are the critical macronutrients, which are known as the most commonly deficient fertilizer elements. Of the macronutrients applied in fertilizers, nitrogen is one of the most important elements in farmland soil. It has more influence on the tree growth, appearance and fruit production/quality than any other element as nitrogen is necessary in the formation of new cells and organic compounds in their structure.

Excess nitrogen application may cause groundwater contamination if leached with excess irrigation or rainfall. Soils with abundant nitrate amounts and shallow groundwater tables pose a high risk to nitrate leaching into drinking water supplies. The greatest concern of nitrate in groundwater is for infants less than 1 year old and for pregnant animals (Killpack, Buchholz *et al* 1993). The nitrate consumed is converted to nitrite which combines with haemoglobin reducing the oxygen-carrying ability of the blood often resulting in death. Therefore there is a need to determine the amount of Nitrogen present in the soil.

The classical analysis methods, namely laboratory analyses for food, soil or plant samples are expensive, time-consuming and require much work first in the collection of samples from fields and second in the laboratory work itself. They also require highly skilled operators and are not easily adapted to on-line monitoring. Related to these disadvantages, sampling and laboratory analysis methods are not effective enough to meet the growing demand of industry.

There are a large number of design concepts available for various soil parameters analysis. Adamchuk V. I *et al* (Adamchuk V. I., Hummel J. W. *et al* 2004)has discussed different sensors used for measurement of different soil parameters. It included following measurement methods:

- Electrical and electromagnetic sensors measure electrical resistivity/conductivity, capacitance or inductance affected by the composition of tested soil.
- Optical and radiometric sensors use electromagnetic waves to detect the level of energy absorbed/reflected by soil particles.
- Mechanical sensors measure forces resulting from a tool engaged with the soil.
- Acoustic sensors quantify the sound produced by a tool interacting with the soil.
- Pneumatic sensors assess the ability to inject air into the soil.

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- Electrochemical sensors use ion-selective membranes that produce a voltage output in response to the activity of selected ions (H^+, K^+, $NO3^-$, Na^+, *etc*.).

Each measurement concept mentioned above, targets only few soil properties. The study shows that prediction of residual nitrate or nitrogen content is possible by spectroscopy which is a spectro-photometric method. NIRS technology has been applied in many areas, such as the food industry, petrochemical industry, and in agriculture, because of its testing speed, low cost, and non-destructive as well as real-time testing (Xiaofei A., Minzan L. *et al* 2013).

It includes several instrumental techniques such as reflectance spectroscopy and fluorescence spectroscopy. Together with fiber optic sensors, they allow on-line monitoring of products in factory or in field as remote or in-situ sensors. Soil spectroscopy has been researched for more than 30 years, and the numbers of papers on visible-NIR in soil science journals has increased exponentially over the last 20 year. Obviously the technique is useful and it can be used to estimate properties such as SOM, mineral composition, clay content and water. As, the spectrum is an integrative property of the soil that tells us about its mineral and organic composition, it could be used directly in soil mapping, for monitoring soil, for making inferences about its quality and function, and making geomorpholocial interpretations of its distribution (Stenberg B., Raphael A. *et al* 2010).

The NIR spectral region has also been used for predicting organic Carbon(C) and total N in soils (Dalal and Henry *et al* 1986; Sudduth *et al* 1991). Zheng et al. (2008, 2009, 2010) estimated soil parameters using NIR reflectance. Similarly, (An *et al* 2011) and (Xiaofei A., Minzan L. *et al* 2012) also successfully developed TN detector model using NIRS.

2. Near Infra-red Spectroscopy (NIRS)

2.1 Overview of NIRS

Near Infrared reflectance spectroscopy (NIRS) is the rapid, non-destructive analytical technique which allows the simultaneous estimation of standard soil characteristics and does not require the use of chemicals. Near infrared spectroscopy is a spectro-photometric method that deals with the interactions of near infrared radiation with the sample under investigation. It is based on the absorption of electromagnetic radiation at wavelengths in the range of 780-2500 nm. It is an analytical technique that characterizes materials according to their absorbance or reflectance in the NIR range(Valerie G., Gilles C *et al* 2011).

The NIR spectral region is dominated by weak overtones and combinations of vibrational bands of light atoms that have strong molecular bonds such as H attached to atoms of N, O or C (Dalal, Henry *et al* 1986). Each constituent of a complex organic mixture has unique absorption properties in the NIR region (780-2500 nm) due to stretching and bending vibrations of molecular bonds between elements (Morra *et al* 1991).

To generate a soil spectrum, radiation containing all relevant frequencies in the particular range is directed to the sample. Depending on the constituents present in the soil the radiation will cause individual molecular bonds to vibrate, either by bending or stretching and they will absorb light to various degrees with a specific energy quantum corresponding to the difference between two energy levels. As the energy quantum is directly related to frequency and inversely related to wavelength, the resulting absorption spectrum produces a characteristic shape that can be used for analytical purposes (Miller *et al* 2001). The frequencies at which light is absorbed appear as a reduced signal of reflected radiation and are displayed in percentage reflectance(R), which can then be transformed to apparent absorbance.

The wavelength at which the absorption takes place (*i.e*., the size of the energy quantum) depends also on the chemical matrix and environmental factors such as neighbouring functional groups and temperature, allowing for the detection of a range of molecules which may contain the same type of bonds (Stenberg B., Raphael A. *et al* 2010).

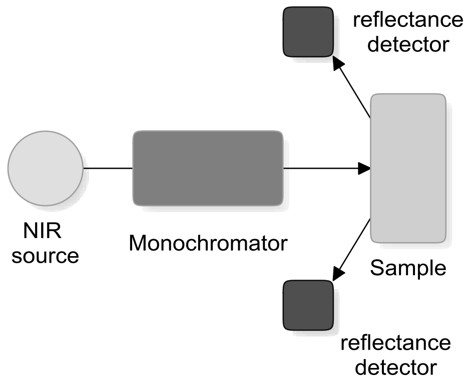

Fig.1 Principle of NIRS technique

As shown, the near infrared radiation penetrates the surface layer of powdered sample and excites molecular vibration and then it is scattered from the sample. In this case, the diffusely reflected radiation (R) is the ratio of the intensity of the radiation reflected from the sample to that of the standard reflector, such as a ceramic disk and it can be converted to absorbance.

2.2 Factors affecting prediction accuracy

It is well known that, soil moisture content and soil particle size are the main factors influencing the measurement accuracy in addition to soil colour. Hence, quantification of the effects of these properties might be useful for the improvement of calibration models. Similarly, spectra pre-processing, sample preparation, size of calibration area, spectrophotometer wavelength range and type of detectors and calibration methods also affect calibration accuracy.

2.3 Soil preparations

Before scanning soil samples, a preparation procedure need to be defined. In order to be used for routine work in laboratories, the procedure should be easy to apply and give similar spectra regardless of the laboratory, the technician or the physical nature of the soil sample. Furthermore, the same spectra scattering should be observed whatever the soil texture. Based on the studies, it was decided to work with 2mm crushed and sieved soil samples. (Valerie G., Gilles C. *et al* 2011)

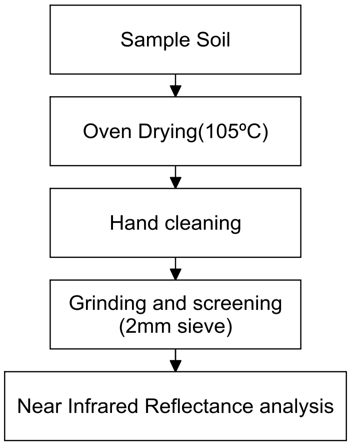

Fig. 2 Sample preparation steps

Barthes *et al* (Barthes *et al* 2006) conducted a research to assess how sample preparation affects NIR prediction of TN and total C. They concluded that the most accurate predictions were obtained with oven-dried finely ground samples, with limited response to sample replication.

2.4 NIR Wavelength determination

Most of the spectroscopic techniques used several kinds of spectrometers to ensure the accuracy of the results. These systems were too expensive to be applied in practice because of the use of spectrometers. Therefore there was a need to find an alternative to spectrophotometer for designing a light, portable and cost effective TN detector model. Zheng et al. (2008, 2009, 2010) tested several algorithms to estimate soil TN by NIR spectral reflectance using MATRIX-I. These algorithms included multiple linear regression, BP-NN, wavelet analysis, and SVM. After comparing the results of these algorithms, Zheng et al. suggested the following 24 wavelengths for soil TN estimation: 2234, 2150, 1991, 1833, 1895, 1684, 1673, 1559, 1536, 1394, 1389, 1311, 1286, 1215, 1208, 1187, 1124, 1092, 1064, 1028, 984, 972, 931, 923, 859, and 844 nm (Xiaofei A., Minzan L. *et al* 2013).

An et al. (2011) further studied the soil TN estimation model and measured the spectral data using MATRIX-I. Several wavelengths combinations were compared to determine an optimal combination. Finally, a new group of wavelengths(1550, 1300, 1200, 1100, 1050, and 940 nm) was suggested. Further Xiaofei An, Minzan L. *et al.* developed successfully a portable soil nitrogen detector based on NIRS using six LEDs corresponding to these six wavelengths. The choice of LEDs solved both the portability limitations. Also, it made the system light weight and commercial.

3. Structure Design

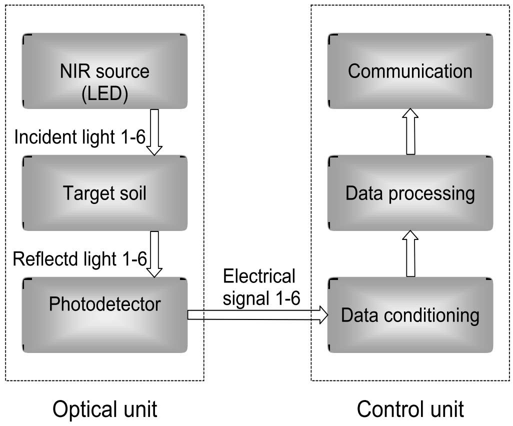

Fig.3 System flow diagram

The detector consists of an optical unit and a control unit. The optical unit includes six near-infrared sources (LED), a medium (optical fiber) for carrying light and a detector. The control unit consists of an amplifier circuit, a filter circuit, an analog-to-digital converter (A/D) circuit, an LCD display. The optical signal at each wavelength is transferred from the LEDs to the surface of the target soil. The reflected light from the soil surface is acquired and transferred to the photodetector, through which the optical signal would be converted to an electrical signal. Subsequently, the electrical signal is amplified to achieve adequate voltage level and amplified signal thus obtained is then digitized using ADC, and the absorbance at each wavelength will be calculated. All six absorbance data will be used as inputs for the soil TN content estimation model. Calibration of the result is done using appropriate multivariate calibration technique, to achieve optimal accuracy. Finally, the calculated soil TN content will be displayed on the LCD display.

Absorbance is selected as the spectral parameter. A standard whiteboard is taken as reference, which is needed to obtain the absorbance of the soil samples. The output voltage Vi is obtained from the reflected rays due to the standard whiteboard. Then, the output voltage V_o corresponding to the required soil sample. The reflectance R_i and absorbance Ai of every soil sample can be calculated according to equations (1) and (2) (Xiaofei A., Minzan L. *et al* 2013).

$$R_i = \frac{V_o}{V_i} \times 100\% \quad (1)$$

$$A_i = \log\left(\frac{1}{R_i}\right) \quad (2)$$

Where, i indicates 1550, 1300, 1200, 1100, 1050 and 940 nm; Vi is the standard whiteboard output voltage at i; V_0 is the soil sample output voltage at i; R_i is the reflectance at i and A_i is the absorbance at i.

4. Multivariate Calibration Techniques

Calibration is the process, which relates the measured analytical signal to the concentration of the analyte. In other words, the idea of a calibration method is to develop a mathematical model between the instrument response and analyte concentration and then to use such model for the prediction of analyte concentrations in the approaching instrumental analyses. Various calibration methods have been used to relate near-infrared spectra with measured properties of materials. The most used multivariate calibration techniques for NIRS are:

1. Principal components regression(PCR)
2. Partial least squares regression (PLSR)
3. Wavelet transform
4. Artificial neural networks

4.1. Principal components regression (PCR)

Principal components regression (PCR) is a two-stage process. First, it minimizes the number of independent components required to describe the variations across the entire spectrum and between spectra. This technique enables several thousand spectral points to be reduced to a few principal components (PCs), where the PCs describe the spectral variance across all the samples. Second, these PCs are regressed against known property data (measured concentration), and then calibration models constructed (Cheng-Wen C., David A. Laird *et al* 2001). These models are validated using separate independent and well-characterized samples to ensure they are robust enough to be used to predict property data from spectral information (Brereton *et al* 1990).

4.2. Partial least squares (PLS)

PLS regression is a bilinear regression process developed by Wold and co-workers (Wold *et al* 1989). It is similar to PCR except that PLS regression uses both the spectral and known property data simultaneously during the calculation of the principal components. The spectral and known property data are projected onto a latent variable, and a second orthogonal variable is derived from the residuals. This process is repeated until the model is complete. The method has advantages in reducing noise, detecting unknown samples that are not represented by the calibration model and eliminated the need for wavelength selection.

4.3. Wavelet analysis

Wavelet analysis was developed to overcome the limitations of the Fourier transform with non-stationary signals and the resolution problems of the STFT. The procedure is similar to Fourier analysis where the signal is multiplied and integrated by a function. However, rather than using sine and cosine functions, wavelet analysis uses scaled and shifted versions of a base function called a mother wavelet. Unlike sine and cosine functions these mother wavelets are local and finite, making them ideal for approximating signals with sharp peaks and discontinuities. Selecting a mother wavelet that represents the general shape of the signal is important (Jahn B.R. and Linker R. *et al* 2006).

The continuous wavelet transform Wf is defined as,

$$Wf(s,\sigma) = \int_{-\infty}^{+\infty} f(t)\frac{1}{\sqrt{|s|}}\psi * \left(\frac{t-\sigma}{s}\right)dt \qquad (3)$$

where, f(t): the time representation of the signal; t : time; * :stands for the complex conjugate.

The continuous wavelet transform of the signal is a function of two factors, 's' and 'σ'. The factor s is the scale factor and causes the mother wavelet to either stretch or dilate. The translation factor 'σ' is related to the location of the window as it is shifted along the signal. The term 'ψ' represents the mother wavelet.

4.4 Artificial neural network modelling

ANNs provide a method to characterize synthetic neurons to solve complex problems in the same manner as the human brain does. Neural networks use machine learning based on the concept of self-adjustment of internal control parameters. An artificial neural network is a non-parametric attempt to model the human brain. Artificial neural networks are pliable mathematical structures that are capable of identifying complex non-linear relationships among input and output data sets. The principal differences between the various types of ANNs are arrangement of neurons and the many ways to assess the weights and functions for inputs and neurons.

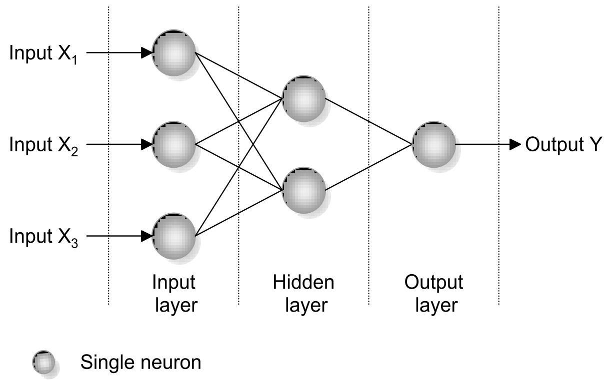

Fig.4 Simple artificial neural network

In case of artificial neuron the information comes at the input layer via inputs that are weighted, *i.e.* each input is individually multiplied with a weight. Neurons, then sums the weighted inputs, bias and process the sum with a transfer function. At the end an artificial neuron passes the processed information via output.
A mathematical description below:

$$Y(k) = \mathrm{F}\left(\sum_{i=0}^{m} w_i(k)\cdot X_i(k) + b\right) \qquad (4)$$

where, $X_i(k)$ is input value in discrete time k; $w_i(k)$ is weight value in discrete time k; b is bias; F is a transfer function; $Y(k)$ is output value in discrete time k.

R^2 is used to compare the prediction accuracy of different models for different properties. It is a statistical measure of how close the data are to the fitted regression line. It is also known as the coefficient of determination. Higher the R-squared, the better the model fits data.

Studies show that, BPNN outperforms over PCR and PLSR (Cheng-Wen Chang and David A. Laird al *et al* 2001). By applying BPNN calibration technique for soil TN content, the calibration R^2 was found to be 0.88 (Xiaofei A., Minzan L. *et al* 2013). Wavelet analysis was applied to soil spectra collected by (Linker *et al.* 2004) and soil nitrate calibration equations were developed. R^2 was found to be greater than 0.9 (Jahn B.R. and Linker R. *et al* 2006). Thus, wavelet analysis is superior over the rest and could provide very accurate results.

Conclusions

Spectroscopy is an emerging technology having vast applications in different fields such as cereal industry, dairy industry, meat industry, *etc.* The property of spectroscopy to characterise the materials according to the reflectance obtained, can be used in precision agriculture to achieve real time information about soil characteristics or soil ingredients. Nitrogen being the key component for plant growth, the knowledge about soil Nitrogen content will be beneficial for farmers to improve crop production.

The accomplishments reviewed in this paper, have led to the modified model consisting of six LEDs corresponding to wavelengths 1550, 1300, 1200, 1100, 1050 and 940 nm, as a NIR light source. Thus, a portable and cost effective TN detector model can be developed, eliminating the need of expensive and bulky spectrometers. Also, it has found that wavelet analysis was used to identify a few wave numbers at which interferences from other ions were minimal. This method produced calibration equations that were soil independent and gave superior results ($R^2 > 0.9$) compared to other methods.

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