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HAL Id: hal-00396317
https://hal.science/hal-00396317

Submitted on 17 Jun 2009

Near-infrared reflectance spectroscopy (NIRS): a practical tool for the assessment of soil carbon and nitrogen budget

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Abstract

The assessment of greenhouse gas emissions from soils requires an accurate knowledge on the fate of carbon (C) and nitrogen (N) in soils. This knowledge shouldn't be limited to C and N stocks in soils but should incorporate the quantification of functional soil organic matter (SOM) fractions with different turnover rates. Conventional analysis can't be used to assess such functional properties of SOM over large geographical areas. Near-infrared reflectance spectroscopy (NIRS) is a rapid and cheap method that could achieve this goal. This technology has proven its efficiency to discriminate among functional SOM fractions and to predict C, N on large sets of soil samples at a large regional scale. NIRS can be used for the detailed analysis of decomposition and aggregation of SOM, two key ecosystem processes that control C and N budget in soils. This review points up the improvement of NIR prediction models by the technique of local calibration and the potential of NIR analysis to assess the ontogeny and heterogeneity of detritus in soils. Mapping soil C, N and SOM fractions at the European level is a challenge that NIRS can help to take up.

Keywords: NIRS; Soil organic matter pools; Soil carbon sequestration; Soil mapping; Ontogeny of detritus; Decomposition; Soil aggregation; Local calibration

Introduction

When trying to assess greenhouse gas budget of soils (*e.g.* Cost Action 639), it is important to get accurate information on carbon stocks (CS) in soils at a global scale. Increasing carbon stocks in soils is possible through changes in land use management practices (Lal, 2004), but enhancing CS in soils can increase emissions of N_2O and CH_4 (Lal, 2004) which are other important greenhouse gases. Monitoring changes in soil CS with precision requires the determination of a baseline value (Lark *et al.*, 2006). At European level, a map of organic carbon in topsoils (Jones *et al.*, 2004) is already available from the European Soil Bureau based at the Joint Research Center (Ispra, Italy). This map defines the baseline status of soil organic carbon (SOC) in 1990. Although this map generated by pedo-transfer rules presents good correlations with measured values, its confidence level remains quite heterogeneous across large areas.

Current models of soil organic matter (SOM) dynamics need more detailed information describing the heterogeneity of SOM: the quantification of SOM pools with different turnover rates (Six *et al.*, 2002). Measuring SOM pools is time-consuming and seems unfeasible with a fine-resolution at a global scale. Thus, there is an urgent need for developing methods for determining SOC and SOM fractions that would allow the analysis of large number of samples necessary for monitoring purposes over large geographical areas.

Near-infrared reflectance spectroscopy (NIRS) is a rapid and non-destructive analytical technique that involves diffuse-reflectance measurement in the near-infrared region (1000 – 2500 nm). Near-infrared spectra (Figure 1) depend on the number and type of chemical bonds in the material being analysed (Foley *et al.*, 1998). NIR spectra can be used to predict various soil properties including C and N content in soils (*e.g.* Chang & Laird, 2002, Ludwig & Khanna, 2001). It has been recognized as a practical indicator and tool to measure soil health and their basic characteristics (Bunning & Jimenez, 2003). With the recent development of *in situ* NIR analysers, NIRS is now considered as a promising emerging technology for measurement of soil C (Gehl & Rice, 2007) in the field.

The aim of this short paper is to briefly review the capacity of NIRS to quantify soil C, N and functional SOM fractions from the field level to a global scale. First, we describe the methodology of NIRS for the analysis of soil samples. Then, we present most recent NIRS researches on the detailed study of two key soil processes controlling C and N stocks in soil: decomposition and aggregation of SOM. In a third part, we review the use of NIR data for soil C and N mapping. We finally list a number of key issues for future development of NIRS in routine analysis of soil C and N budget.

1. Implementation of NIRS in Soil Science

Sample processing for NIR analysis of soil is rather simple. Soil samples can be either air-dried or oven-dried at 40°C. Particle size and moisture content influence NIR spectra (Barthès *et al.*, 2006). Thus, soil samples are dry-sieved (2 mm), ground (0.25 mm) to obtain a homogeneous powder and stored in polypropylene boxes. The day before NIR analysis, powders are oven-dried at 40°C during the night to homogenize moisture and kept in a desiccator before analysis. From 1 g to 25 g of soil is required for NIR analysis, depending on the NIR analyser. Spectra consist of absorbance (absorbance = log [1 / reflectance]) values from 1000 to 2500 nm. From 100 to 300 samples per day can be scanned.

The diffuse-reflectance spectra of soil result from bending, twisting, stretching or scissoring of different chemical bounds (*e.g.* C–H, O–H, N–H) under near infrared radiation (Ludwig & Khanna, 2001). Absorptions in the near-infrared are weak since they consists of overtones or combinations of fundamentals (Wetzel, 1983). Direct interpretation of NIR spectra is almost impossible : each NIR analysis require a statistical treatment. First, spectral data should be normalized with a mean centering technique and can be variance scaled. Then, the first or second derivative of spectra can be computed to reveal information hidden in the spectra, and also to reduce baseline drift. Smoothing the optical signal with derivative filters (mainly Norris or Savitzky-Golay) can help to lessen spectral noise as well as scattering corrections like multiplicative scatter

correction (MSC) or standard normal variate (SNV) correction. After pretreatment, statistical tools relevant to the aim of the analysis are implemented to analyse spectral data.

NIR analysis of soil can be run in two main ways (Figure 2): (i) qualitative or (ii) quantitative. (i) Qualitative analysis of soil with NIRS can be used to discriminate among soil samples from a given population with principal component analysis of soil spectra (PCA) or redundancy analysis (RDA), build classes of soil samples with discriminant analysis (DA) or to study time series of soil samples under controlled conditions.

(ii) Quantitative analysis allows the prediction of various components and soil properties after a calibration of NIR spectra against reference values. Quantification with NIRS usually involves multivariate techniques (*e.g.* partial least square regression, PLS) that consist of regression method used to build prediction models (Dardenne *et al.*, 2000). Recently, a local approach for calibration of NIR prediction models (Brunet *et al.*, 2007, Dardenne *et al.*, 2000, Genot *et al.*, 2007, van Waes *et al.*, 2005) has attracted much attention. Local calibrations are carried out within spectral classes defined either on statistical distances among spectra (Mahalanobis distances) or directly upon soil types, texture groups or land use.

The quality of NIR prediction models is assessed with various indices (Fearn, 2002, Williams, 2001):

- SECV: standard error of cross-validation performed on the calibration data set
- SEP: standard error of prediction which is the standard deviation (SD) of differences between NIR reflectance and reference values, that should be calculated on real independent data set, but is usually calculated on a non independent validation data set
- RPD: the ratio of performance to deviation, (SD of reference values) / SEP
- R^2: the coefficient of determination

2. Use of NIRS to study decomposition and aggregation of SOM

The amount of carbon returned to the atmosphere by decomposition of dead organic matter (OM) is a key component of the global carbon budget (Aerts, 1997). SOM decline is one of the major threat regarding soil degradation in Europe (Bellamy *et al.*, 2005, SEC(2006)1165). During soil formation, organisms create and manipulate detritus into complex SOM pools that contribute to OM protection against decomposition (*e.g.* Lavelle & Spain, 2001, Lützow *et al.*, 2006). Biological and physical aggregation of OM is one of the main process responsible for the creation of such SOM pools with different turnover rates (*e.g.* Blanco-Canqui & Lal, 2004). This heterogeneous nature of detritus that results from variation in sources, and the changes it undergoes during decomposition has recently been incorporated in a conceptual model of ontogeny of detritus by Moore *et al.* (2004). Finding an operational expression of the heterogeneity and ontogeny of detritus (Moore *et al.*, 2004) that could easily quantify functional SOM pools (Blanco-Canqui & Lal, 2004, Robert *et al.*, 2004) is a challenge for soil scientists.

2.1 Decomposition process

The suitability of NIRS in decomposition studies has been demonstrated by Joffre *et al.* (1992) for plant litter materials. Litter decomposability can be predicted with NIRS (Gillon *et al.*, 1999). It is also possible to discriminate among soil organic layers, predict their biological and chemical properties (Chodak *et al.*, 2002, Ludwig *et al.*, 2002) like C, N and SOM fractions including microbial biomass at different decomposition stages with this technology (Coûteaux *et al.*, 2003). It is also possible to predict the relative amount and the composition of litter and root mixtures with NIRS (M.C. Gruselle, unpublished PhD, Roumet *et al.*, 2006). Recently, Cécillon *et al.* (2007) have shown with NIRS that a holistic approach of decomposition is possible in order to obtain real-world decay constants for organic horizons of mix-stands. With a PCA of NIR spectra, it is possible to follow decomposition trajectories of detritus under different experimental conditions. The effect of soil mesofauna on litter decomposition can be quantified with PCA (Figure 3) which is the first example of a preliminary exploration of the ontogeny of detritus with NIRS.

2.2 Aggregation process

Soil invertebrates produce compact organic and organo-mineral faecal pellets (Lavelle & Spain, 2001). These animal faeces, which are major components of humic material can form stable SOM pools (Rawlins *et al.*, 2007). Hedde *et al.* (2005) have shown with PCA of NIR spectra that each soil biogenic structure has a specific NIRS signature, different from the surrounding soil. A change in decomposability of organic matter from leaves to faecal pellets of soil fauna has been revealed by NIRS (Gillon & David, 2001). Recently, Velasquez *et al.* (2007) have classified soil aggregates upon their origin (biogenic, physicogenic ; Figure 4) with PCA analysis of NIR spectra. A current research work at Cemagref Grenoble has shown a change in the NIR spectra of soil and biogenic structures of earthworms after forest wildfires (Master thesis of S. Figarol, IRISE project, Forest Focus). Spectral differences between soil and biogenic structures are reduced in plots impacted by recent wildfires.

All these NIRS studies show the potential of this technology for the study of macroscopic functional SOM pools. But current SOM models need the quantification of microscopic functional SOM pools, as a recalcitrant SOM pool as been identified in microaggregates within soil macroaggregates (Six *et al.*, 2002). These microscopic functional SOM fractions can be quantified with NIRS (Cozzolino & Moron, 2006) as well as their respective C, N content and mineralization rates (Mutuo *et al.*, 2006). However, prediction of these functional SOM pools with NIRS have to be confirmed on larger data sets, at the regional scale.

Finally, the power of NIRS in SOM studies have been demonstrated by Joffre *et al.* (2001) who managed to link experimental NIR spectra to SOM theoretical quality, q (Q model, Ågren & Bosatta, 1998).

3. Mapping of soil C and N with NIRS

Mapping soil C and N budget of soil at a regional scale implies analysing large number of samples. It has been estimated that monitoring soil C in all US farms, with existing analytical techniques would cost more than $1 billion for the first 4-yr period (Subak, 2000). A reduction in per-sample

cost analysis for soil C and N, coupled with a quicker sampling and sample preparation method would be welcome (Martin *et al.*, 2002). That's why NIRS has recently been tested for mapping soil C and N at the field and regional scale.

At the field level, Martin *et al.* (2002) found useful correlations for SOC using PCA and PLS regression of NIR spectra. However, they failed at predicting soil organic nitrogen with the same experimental design. Their conclusion is that NIRS has potential to be a suitable method for SOC mapping for the purposes of monitoring C sequestration. A study of Yong *et al.* (2005) has shown good correlations between a map of SOM predicted by NIRS and a map of SOM measured with a conventional method. This research found also a good prediction of total soil N with NIRS. Odlare et al. (2005), used a NIR-PCA strategy to determine soil spatial variation in a field. They compared maps of soil properties with maps of PCA scores of NIR spectra and concluded that this technique can be useful in precision farming.

At the regional scale, a recent study of Genot *et al.* (2007) used NIRS to predict soil properties including SOC on a large data set of 946 samples representative of the soil diversity of the Wallonia region (Belgium). They tested various calibration method (global and two local calibration techniques) and found that local calibrations usually improve the quality of NIR prediction models (reduction of SECV).

Prediction of SOM at the global level could be facilitated by the use of portable NIR analyser. Gehl and Rice (2007) have classified NIRS among emerging technologies for *in situ* assessment of soil carbon. But the quality of NIR measurements in the field is still in debate, and NIR laboratory measurements are very rapid and simple. Maybe linking NIR field measurements to NIR remote sensing measurements from aircraft could be a suitable way of mapping soil properties over large geographical areas (Lagacherie *et al.*, 2007). Another promising approach for mapping soil properties is to use predictions made with spectral data in inference systems to predict and map other important soil properties (McBratney *et al.*, 2006).

A drawback of most studies is that prediction capacity of NIRS is usually not tested on a real independent data set of soil samples. A research project carried out within the framework of Cost Action 639 – BurnOut will test the suitability of NIRS for SOC mapping on independent data at

the field scale (Short Term Scientific Mission – STSM of L. Cécillon at Cranfield University, UK, with P. Bellamy). Another promising way of mapping SOM could be the use of NIR spectra (PCA scores) of topsoil as secondary information to improve CS maps (*e.g.* Simbahan *et al.*, 2006). This approach will also be tested during the same forthcoming STSM within Cost Action 639.

4. Future directions for NIRS studies in Soil Science

Diffuse reflectance spectroscopic techniques have a real potential for analysing simultaneously various soil properties on large number of samples (Viscarra Rossel *et al.*, 2006). In this review and another recent paper of Reeves *et al.* (2006), the quality of NIR prediction models is compared to mid-infrared technology (2500 – 25000 nm). These two studies found that both technologies have potential for measuring soil properties but they underlined that MIR predictions were sometimes better. However, these researches didn't use the promising local calibration technique that can be decisive for the improvement of NIR calibration models (Genot *et al.*, 2007).

Although we pointed out the need for an assessment of prediction capacity of NIRS on independent data, we strongly believe that one of the most important topic in NIRS studies is the generalization of local calibration. That implies building large database of spectral data representative of soil diversity in Europe that could be shared between laboratories. To achieve this goal properly, the processing of samples (sieving, grinding, oven-drying and scanning) should be standardized to ensure that spectral data are comparable among laboratories. All promising techniques described in this paper suffer from the small number of samples used for calibration and validation of NIR prediction models, the lack of studies at a regional or European level and the use of the global calibration method which is generally not the best way in soil studies. Thus, a key issue would be to test their suitability over large areas using local calibration.

Regarding the assessment of greenhouse gas emissions from European soils which is the aim of Cost Action 639 – BurnOut, a challenge of NIRS studies is the quantification of functional SOM pools. Such NIR prediction models should be validated over large geographical areas, with a fine-resolution and directly interoperable with global models of C and N cycles.

Acknowledgments

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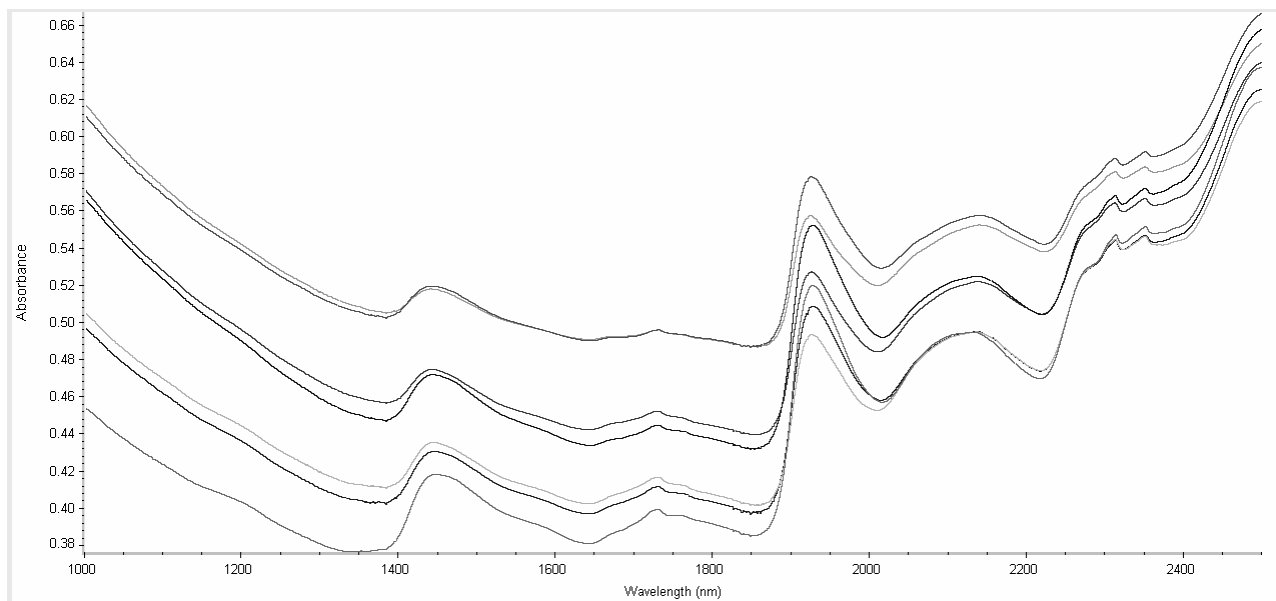
Figure 1: Examples of NIR spectra of forest topsoils –

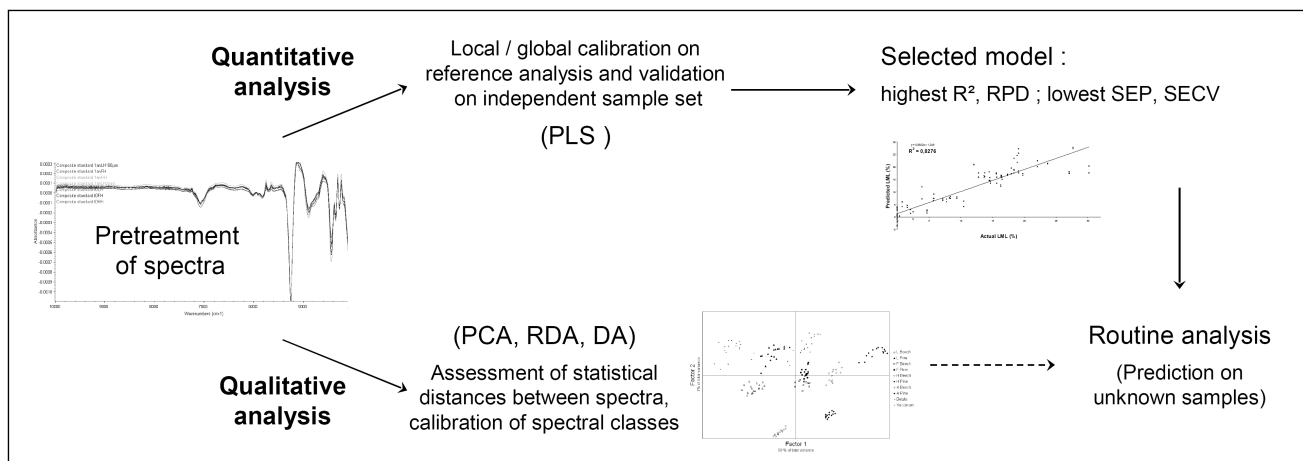Data acquisition with an Antaris II NIR Analyser (Thermo electron corporation)

Figure 2: Quantitative and qualitative analysis of NIR spectra
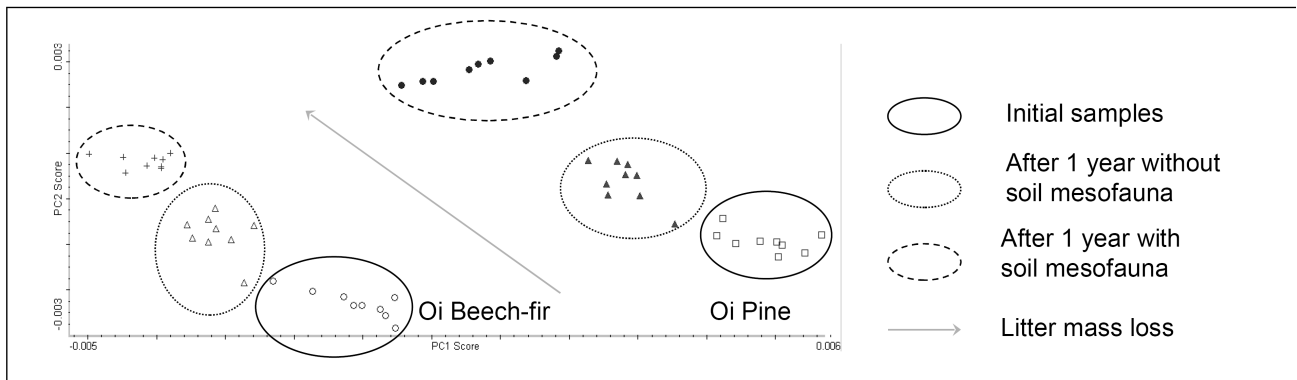
Figure 3: Impact of soil mesofauna on the ontogeny of forest litter
(PCA of NIR spectra; PC1: 72% of total variance, PC2: 25% of total variance)
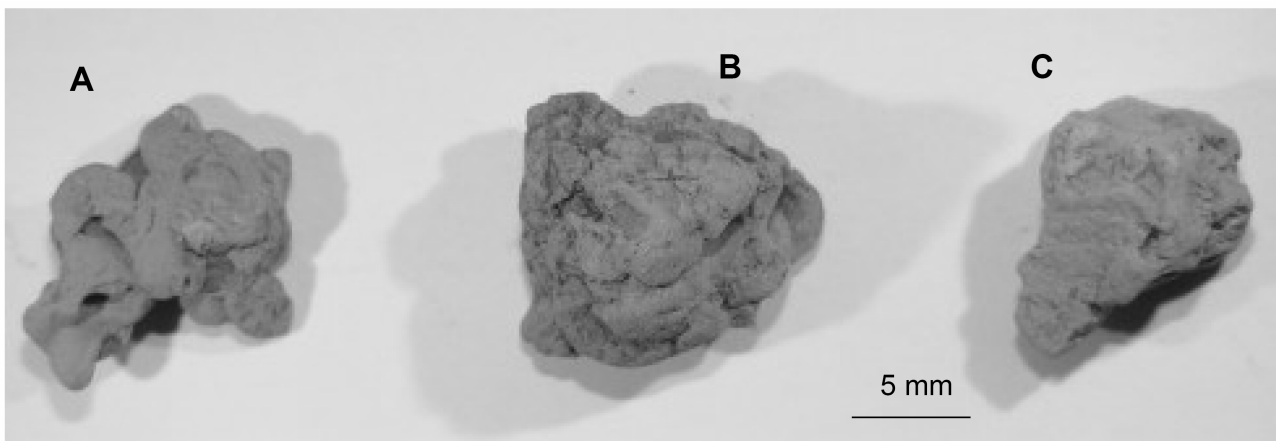

Figure 4: Soil macroaggregates
(A) Biogenic
(B) Intermediate
(C) Physicogenic