2007

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Recommended Citation

Flicklin, Robert L.; Foley, Shane M.; and Kissell, Robert E. Jr. (2007) "Quantifying Soil Chemical Properties Using Near Infrared Reflectance Spectroscopy," *Journal of the Arkansas Academy of Science*: Vol. 61 , Article 8.
Available at: http://scholarworks.uark.edu/jaas/vol61/iss1/8

# Quantifying Soil Chemical Properties Using Near Infrared Reflectance Spectroscopy

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*Abstract.*—Methodologies for determining soil chemical properties have evolved dramatically during the past century. Early geochemical analyses were conducted exclusively through the use of wet chemistry techniques that were relatively reliable but painstaking and subject to errors at various stages of analysis. Near infrared reflectance spectroscopy (NIRS) has emerged as a new approach for rapidly analyzing a variety of materials including soils. In this study soil samples were taken from eight study areas across the Ozark Highlands of Arkansas, and NIRS calibration models were developed to determine the accuracy of using NIRS to analyze soils compared with standard soil chemical analysis protocols. Multivariate regression models were highly effective for analyzing several important elements. C and N models explained 92% and 88% of their variation, respectively, and Ca, Mg, P, and Mn models explained 72-88% of the variability in these elements. Models for C:N and pH explained 82% and 86% of their variability, respectively. Models for micronutrients Cu and Zn did not fit as well with 22% and 40% of their variability explained, respectively. Our findings suggest that additional NIRS calibration and modeling is promising for rapidly analyzing the chemical composition of soils, and it is desirable to develop model libraries that are calibrated for the soils of a given region.

*Key words:*—Near infrared reflectance spectroscopy, NIRS, Ozark Highlands, Arkansas, soil chemical properties.

## Introduction

Agricultural and silvicultural production has been revolutionized by a broad array of technical advances that continue to elucidate the biochemical and biogeochemical processes underlying vegetative growth in natural and managed environmental systems. Prior to the work of Liebig (1843) and his contemporaries, supplementing soils to increase plant vigor and growth was as much art as science. Subsequent research identifying plant essential nutrients coupled with the Haber-Bosch process for synthesizing ammonium from plant-unavailable atmospheric nitrogen continued to improve the ability of land managers to increase biomass production in a variety of ecosystems (Smil 2001). At present scientists and managers are faced with the challenge of continuing the growth of biomass production while simultaneously preserving or improving ecosystem services (Carpenter et al. 2006); however, traditional soil and plant analytical techniques are too time consuming and expensive to implement across the large land expanses necessary for truly optimized ecosystem management.

The emergence of near infrared spectroscopic analytical techniques coupled with powerful multivariate analysis procedures is providing the opportunity to rapidly and accurately provide the same information as traditional laboratory techniques without the cost and potentially hazardous chemical analyses. Over the last two decades, near infrared reflectance spectroscopy (NIRS) has become a well-known and effective analytical tool in agricultural and ecological research, but applications to soil analysis are a relatively recent development (Malley et al. 2004). NIRS has been used for the evaluation of forage nutrition and digestibility (McIlwee et al. 2001), soil quality (Chang et al. 2001), and fecal analysis (Landau et al. 2006). The use of

NIRS has increased because it is a quick, accurate, and cost-effective way to non-destructively analyze samples. NIRS quantifies chemical and physical components by determining the absorption of near infrared radiation by chemical bonds found in sample materials (Dryden 2003). The absorption signatures from spectral scans are used to develop mathematical equations that quantify chemical and elemental components.

The near infrared (NIR) spectrum consists of electromagnetic radiation of wavelengths just beyond the red band of visible light, and the wavelengths of primary inferential importance lie between 800 and 2500 nm (Stuth et al. 2003). Although this band of radiation is not visible to the human eye and is not particularly useful for plant primary production, the photons in this energy range constantly interact with the surfaces of all materials that they contact. When a photon strikes a chemical bond, that photon is absorbed, reflected, or transmitted. If the photons are absorbed by organic molecules, the photons cause the molecular chemical bonds to vibrate and stretch (Gillon et al. 1999). The bonds commonly stretched are C-H, N-H, O-H, C-O, and C-C, although electromagnetic radiation differentially influences all bonds (Stuth et al. 2003). The magnitude of NIR absorption by sample materials is a function of many factors, including intramolecular bonds, molecular orientations, and interactions with neighboring elements (Workman and Shenk 2004). Each bond has a specific range of wavelengths that are absorbed or reflected (Dryden 2003). The affected bonds are abundant in a variety of soil and plant molecules, and new applications of NIRS technology are rapidly developing, particularly in biological and ecological disciplines (Workman and Shenk 2004).

NIR spectrometers utilize sensors that measure wavelengths of radiation that are being reflected by an object to facilitate inferences about the radiation absorbed. The important diagnostic

information for statistical modeling is in the wavelengths absorbed instead of the wavelengths reflected (Locher et al. 2005) since this absorption of radiation allows for bond identification and quantification. To measure absorption spectrometers must either emit a known quantity of NIR radiation or measure the radiance of available NIR radiation (Foley et al. 1998). Given a known quantity of NIR radiation emitted, sensors then detect the wavelength and quantity of electromagnetic radiation being absorbed. Once absorption has been quantified for a given material, chemical content is determined through the use of reference samples of known chemical composition (Dryden 2003). NIRS is a powerful analytical methodology, but careful and consistent sample preparation is required for reliable analysis of unknown samples for comparison with reference samples (Alomar et al. 2003). Moisture content, particle size, drying temperature, sample temperature, and sample packing density can affect the accuracy of spectral analyses (Dryden 2003).

NIRS is a desirable analytical methodology for several reasons, including a short analysis time, the lack of a need for chemical processing, the relatively small amount of sample material needed for analysis, and the ability to preserve the sample after analysis for additional testing if necessary (Foley et al. 1998). Arguably the ability to complete elemental analyses without the use of chemicals is the most appealing reason for adopting NIRS (Dryden 2003). However, the need for only a small amount of sample to achieve accurate results following calibration makes NIRS a good option when insufficient sample material is available to conduct a battery of standard chemical analytical tests.

The purpose of this paper is to evaluate the accuracy of quantifying a variety of soil chemical properties using spectroscopic data from scans of highly weathered Ozark Highland soils by comparison to analytical results from standardized laboratory soil analysis techniques. Specific objectives are to

1) Quantify the ability of NIRS to accurately predict the elemental composition of forest soils from the Ozark Highlands and
2) Quantify the ability of NIRS to accurately predict non-elemental chemical properties of forest soils from the Ozark Highlands.



**Study Sites**

- **National Forest Land**
  - BM- Boston Mountains
  - CS- Ozark National Forest (Cass)
  - SYL- Ozark National Forest (Sylamore)
  - WS- Ozark National Forest (Witt Springs)

- **Wildlife Management Area**
  - GM- Gulf Mountain
  - GR- Gene Rush
  - HEA- Harold E. Alexander
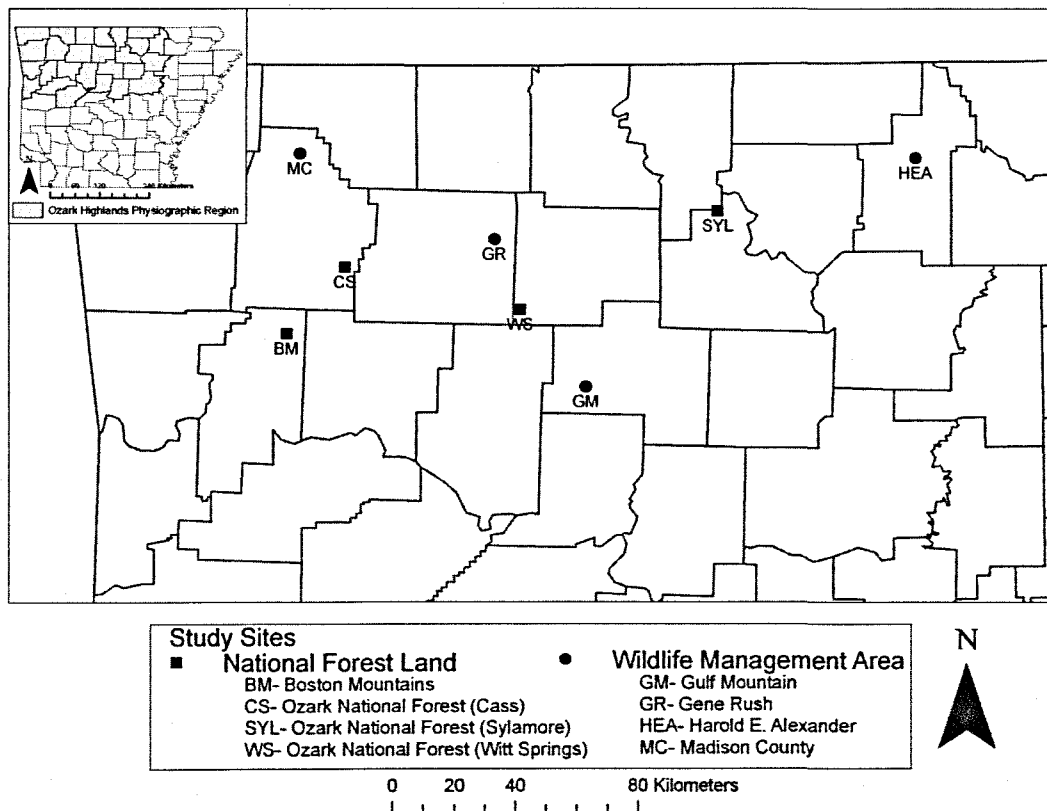  - MC- Madison County

0    20    40        80 Kilometers

N

Fig. 1. Location of study sites on national forest lands and wildlife management areas across the Ozark Highland physiographic region of Arkansas.

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## Materials and Methods

Eight study sites were sampled on public forest lands across the Ozark Highland physiographic region of Arkansas (Fig. 1). Four study sites each were selected from both state lands (Wildlife Management Areas- WMAs) and federal lands (National Forests), so that potential differences in soil chemistry due to management practices would be equally represented in the soil data. Ten soil samples were taken along a transect that conformed to the summit/ridge landforms of each site. Depth of sampling was 15 cm with a minimum distance of 30 m between sample locations. Sampling was restricted to summit/ridge landscape positions to minimize effects on the measured soil properties from differences in landforms across sites (Burrough 1991; Daniels and Hammer 1992). To capture potential seasonal variability of soil chemical properties, each site was sampled during spring, summer, and fall seasons for a total of three sampling periods per site. Sample locations within the study sites were varied each season to avoid the potential influence of prior sampling on the soil chemical properties of interest. The soils across each of the study areas are mapped as Ultisols with high coarse fragment contents. (Soil Survey Staff 2007). The optimal number of samples for the development of calibration equations varies by the type of materials being scanned, but previous research of soil and mineral substrates suggest that 114 (Danieli et al. 2004) to 262 samples (Cozzolino and Moron 2004) provide consistent, robust modeling results. In this investigation 215 soil samples were both chemically and spectrophometrically analyzed. Soil samples were taken in conjunction with plant foliar samples as part of a larger study, and the limited availability/presence of the target plant during the fall of 2005 reduced overall number of soil samples from 240.

Prior to NIRS analysis soil samples were dried at 70°C for 24 hrs to minimize the presence of O-H bonds in water, which can absorb photons over a wide range of wavelengths and hide absorption by other bonds (Foley et al. 1998; Alomer et al. 2003). Soils were passed through a 2-mm sieve using a RoTap® sieve shaker to minimize inconsistent reflectance resulting from path length variance through which electromagnetic radiation traveled due to variable particle size (Foley et al. 1998). Two grams of soil then were loaded into sample cups for spectroscopic analysis with a NIRS Model 4500 Foss-NIRSystems spectrophotometer (NIRS Model 4500 Foss-NIRSystems, Silver Spring, MD). Spectra from 1300-2400 nm were collected during scanning, and the spectra were saved for use in developing calibration equations in combination with the standard laboratory test results of the soil analyses.

NIRS calibration was conducted with ISI software version 3.10 (Infrasoft International, Port Matilda, PA). Spectra were normalized based on their individual Mahalanobis H distance from the mean spectra at each wavelength, which facilitated the identification of spectral outliers for removal. Means of multiple spectral scans were calculated to derive an accurate database of the wavelengths and the quantities absorbed as a way of determining the chemical composition of the soil samples spectrophometrically against which standard soil chemical data were regressed. Spectral absorption graphs were generated to identify diagnostic absorption peaks following mathematical treatments including both first and second order derivatives (Dryden 2003; Danieli et al. 2004; Locher et al. 2005). To optimize the multivariate calibration models, mean centering was performed according to the protocols of Duckworth (2004) to reduce extraneous data and maximize differences between samples of spectral data.

Soil pH was determined using a 1:2 soil:deionized water slurry, and elemental analyses were conducted using inductively coupled plasma (ICP) spectroscopic analysis (Soltanpour et al. 1996) following a Mehlich III extraction (Mehlich 1984). Calibration equations were developed by regressing spectral absorption peaks against the chemical properties measured by standard laboratory methods using three multivariate regression procedures, and the procedure that resulted in the best fit models and cross validation results was selected. Regression techniques utilized were principal component regression (PCR), partial least squares (PLS) regression, and modified partial least squares (MPLS) regression (Gillon 1999; Danieli et al. 2004). Standard error of cross validation (SECV) and Standard error of calibration (SEC) coupled with smoothing treatments were utilized during model selection to optimize model performance while controlling both underfitting and overfitting of the models (Westerhaus et al. 2004).

## Results and Discussion

Model results were mixed with respect to the ability to accurately quantify the full spectrum of soil chemical properties for the Ozark soil samples. Quantification of micronutrient metals copper and zinc through multivariate regression modeling of NIRS data was the least effective with only 23% and 40%, respectively, of the variation explained by the models (Table 1). Calcium and magnesium quantities were predicted very well with 88% and 76% of their respective variances explained. Our results for the Ozark soils are similar to those of Chang et al. (2001) in a study conducted across the western United States in which models for Fe, K, Ca, and Mg quantities explained 50% to 80% of the variance and more than 80% of the variation in C and N compared with chemical analyses. Moreover, NIRS model results for the Ozark soils were very effective for determining quantities of both C and for N with 82% and 88% of their variability explained, respectively. Another study of NIRS with soils from across Africa also found similar predictive ability for determining the quantities of Ca, Mg, and C, as well as pH (Shepherd and Walsh 2002), indicating that NIRS has the potential to be a robust analytical tool for soil elemental analyses when properly calibrated.

A point worth noting is that in the work by Chang et al.

Table 1. Summary of regression methods utilized for model development, mathematical treatments, coefficients of determination, and model validation analytics for selected soil chemical properties.

| Soil Variable | Mean[1] | Statistical Method[2] | Derivative | $R^2$ | SEC[3] | SECV[4] |
|---|---|---|---|---|---|---|
| N | 0.087 | PLS | 1 | 0.881 | 0.011 | 0.013 |
| C | 1.590 | MPLS | 1 | 0.915 | 0.168 | 0.188 |
| C:N | 18.556 | PLS | 1 | 0.820 | 1.408 | 1.635 |
| $NH_4^+$ | 6.095 | PLS | 1 | 0.506 | 1.868 | 2.187 |
| pH | 5.384 | PLS | 1 | 0.857 | 0.132 | 0.160 |
| P | 5.480 | PLS | 1 | 0.715 | 1.178 | 1.282 |
| K | 69.882 | PLS | 1 | 0.609 | 13.706 | 15.554 |
| Ca | 256.122 | PLS | 1 | 0.877 | 73.516 | 90.478 |
| Mg | 47.892 | PLS | 1 | 0.763 | 10.252 | 12.236 |
| S | 8.167 | MPLS | 1 | 0.596 | 1.375 | 1.546 |
| Na | 5.094 | PLS | 1 | 0.413 | 0.615 | 0.664 |
| Fe | 100.350 | PLS | 1 | 0.567 | 13.999 | 15.930 |
| Mn | 95.252 | PLS | 1 | 0.779 | 26.044 | 31.098 |
| Zn | 2.192 | MPLS | 2 | 0.402 | 0.569 | 0.602 |
| Cu | 4.233 | MPLS | 2 | 0.227 | 1.468 | 1.625 |

[1]Units are g kg$^{-1}$ for N and C; all other units are expressed as mg kg$^{-1}$. [2]Multivariate regression procedures: partial least squares (PLS), modified partial least squares (MPLS); [3]Standard error of calibration (SEC); [4]Standard error of cross validation (SECV).

(2001), PCR models produced the most robust models for predicting a range of soil chemical properties. In our study of the Ozark soils, we found that PLS and MPLS models produced superior results compared with PCR models. Since no biogeochemical process is expected to influence the efficacy of one modeling methodology relative to another, our findings suggest that it is appropriate to try all available approaches for which underlying statistical assumptions are met for the purpose of deriving the best model for a particular soil property formed in a particular parent material.

Soil pH is considered a "master variable" for soil chemical properties due to the influence that pH has both on the availability of mineral nutrients and on the activity and composition of soil microbial populations (Rengel 2002). Due to the importance of pH for the management of soils for productivity and potentially for ecosystem services, the ability to assess pH across large areas with good resolution through the analysis of a large number of samples is desirable, and NIRS analysis has been shown to effectively and accurately determine soil pH for a variety of soils. PLS models developed for the Ozark forest soils explained 86% of the variance in pH (Fig. 2; Table 1) from standard measurement techniques. These results are better than those of Chang et al. (2001) and marginally better than the findings of Shepherd and Walsh (2002). However, Chang et al. (2001) extracted samples

across four Major Land Resource Areas (MLRAs), and Shepherd and Walsh extracted samples from an entire continent, whereas our investigation encompassed a physiographic region within one state.

Another soil parameter of ecological importance is the ratio of C to N (C:N). While other studies involving NIRS of soils almost universally assess the ability of NIRS to predict soil C and N independently, no published studies have examined the potential for NIRS to directly measure soil C:N ratio. Soil C:N ratio has profound effects on the stability of soil carbon pools and rates of soil C enrichment/depletion (Lal 2001a; Melillo 2002), and the potential of soils to sequester large amounts of atmospheric carbon to offset greenhouse gas emissions (Lal et al. 1998; Lal 2001b) makes it desirable to have information on the C:N status of soils across large geographic areas. However, if NIRS models are used to determine the concentration of soil C and N independently to derive the ratio of C to N, then the error of prediction for both elements will be compounded. Direct measurement of C:N avoids the error inflation issue, and our model explained 82% of the variation in C:N for the Ozark soils (Fig. 2). While there is variability in model performance, it is clear that NIRS modeling is highly effective for quantifying a number of non-elemental soil chemical parameters across a broad range of soils. Collectively, our results and the results of

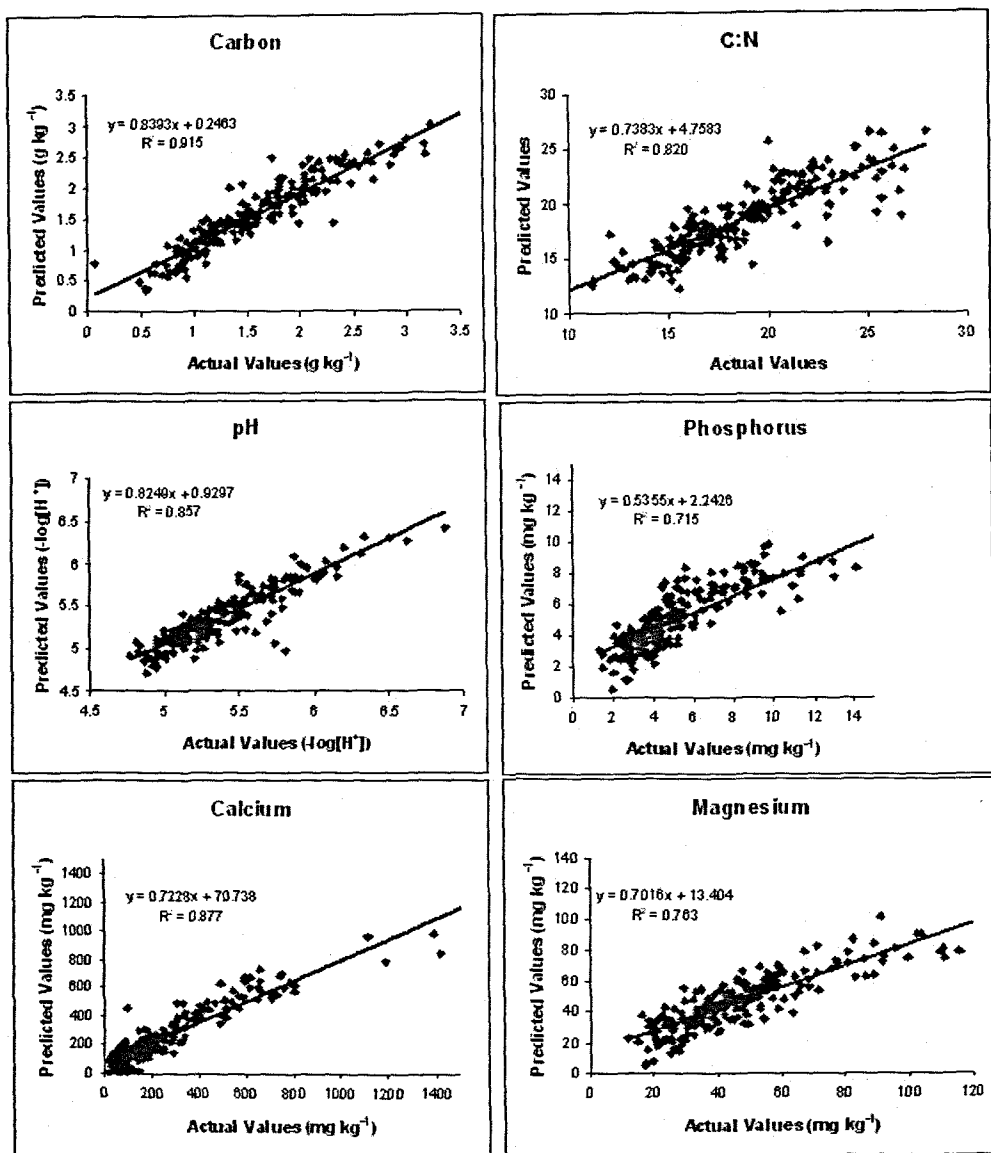Robert L. Ficklin, Shane M. Foley, Robert E. Kissell, Jr.



Fig. 2. Actual values from standard laboratory chemical analyses for select soil chemical properties compared with predicted values using NIRS and multivariate calibration models.

other studies suggest that additional work should be conducted to evaluate the potential of NIRS for quantifying soil properties that are both important for biomass production and ecosystem services and more complex than individual elemental analyses.

Clearly, since models developed in each of the cited studies differ with respect to the spectral data included in the models as well as the mathematical treatments and the modeling methodologies, it is inappropriate to universally apply spectral data scanned using one type of machine under a given set of conditions to any other published model. To assure reliable and accurate results, spectral libraries must be developed using one set of procedures, and all soil samples for which analyses are needed must be processed following clearly defined and reproducible protocols.

## Conclusions

Our results demonstrate that NIRS analytical methodologies can provide a rapid, cost-effective, and robust means by which

land managers can accurately quantify soil quality with respect to potential productivity and ecosystem services. Our findings show that NIRS is a viable analytical approach for quantifying most of the elemental components of Ozark forest soils. Additionally, NIRS also is highly effective for quantifying other soil chemical properties, such as pH and C:N ratio, which are of profound significance for interpreting soil test results and estimating the potential mineralization or immobilization of plant essential elements. Through additional investigation it should be possible to quantify other soil chemical and physical properties with NIRS to glean additional information that can improve both biomass production and ecosystem services.

With increasing pressures globally to increase food and fiber production while also preserving or improving ecological services, analytical tools like NIRS are needed in order for precision resource management to be viable. Once a reference library of spectral data is established, subsequent soil samples can be processed for little more than the cost of sample collection and preparation. Additionally, as the size of the reference library increases, the ability to create robust, accurate models of soil chemical properties also is expected to improve. Advances in spectral analytical technologies are progressing in many disciplines, and it is both wise and appropriate for soil scientists, ecologists, foresters, and biologists to explore the potential of NIRS for advancing our understanding of the processes that drive our natural systems.

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