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Mid-Infrared and Near-Infrared Diffuse Reflectance Spectroscopy for Soil Carbon Measurement

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ABSTRACT

The ability to inventory soil C on landscapes is limited by the ability to rapidly measure soil C. Diffuse reflectance spectroscopic analysis in the near-infrared (NIR, 400–2500 nm) and mid-infrared (MIR, 2500–25 000 nm) regions provides means for measurement of soil C. To assess the utility of spectroscopy for soil C analysis, we compared the ability to obtain information from these spectral regions to quantify total, organic, and inorganic C in samples representing 14 soil series collected over a large region in the west central United States. The soils temperature regimes ranged from thermic to frigid and the soil moisture regimes from udic to aridic. The soils ranged considerably in organic (0.23–98 g C kg⁻¹) and inorganic C content (0.0–65.4 g CO₃-C kg⁻¹). These soil samples were analyzed with and without an acid treatment for removal of CO₃. Both spectral regions contained substantial information on organic and inorganic C in soils studied and MIR analysis substantially outperformed NIR. The superior performance of the MIR region likely reflects higher quality of information for soil C in this region. The spectral signature of inorganic C was very strong relative to soil organic C. The presence of CO₃ reduced ability to quantify organic C using MIR as indicated by improved ability to measure organic C in acidified soil samples. The ability of MIR spectroscopy to quantify C in diverse soils collected over a large geographic region indicated that regional calibrations are feasible.

INCREASING CO₂ CONTENT of the atmosphere from anthropogenic sources has stimulated research to assess the role of terrestrial ecosystems in the global C cycle. The terrestrial biosphere is an important component of the global C budget, but estimates of C sequestration in terrestrial ecosystems are partly constrained by the limited ability to assess dynamics in soil C storage. Agricultural croplands have a great potential for sequestering atmospheric C (Lal et al., 1998), but current technologies for monitoring soil C sequestration in terrestrial ecosystems are not cost effective, or they depend on intensive methods.

Diffuse reflectance spectroscopy offers a nondestructive means for measurement of C in soils based on the reflectance spectra of illuminated soil. Both the NIR (400–2500 nm) and MIR (2500–25 000 nm) region have been investigated for utility in quantifying soil C (Dalal and Henry, 1986; Meyer, 1989; Janik et al., 1998; Reeves et al., 1999; McCarty and Reeves, 2000; Reeves et al., 2001). The characteristics of spectra obtained in these regions varies markedly, with the MIR region dominated by intense vibration fundamentals, whereas the NIR region is dominated by much weaker and broader signals from vibration overtones and combination bands. These divergent spectral characteristics may be expected to have substantial influence on the ability to obtain quantitative information from spectral data.

Over the last two decades, NIR spectroscopy (NIRS) has developed as a major tool for quantitative determinations of components within often complex organic matrices whereas MIR spectroscopy (MIRS) has been used mainly in research for qualitative analysis involving spectral interpretation of chemical structures. The main reason for the exclusion of MIRS in quantitative analysis has been the belief that quantitative analysis using the MIR region required KBr dilution because of the strong absorptions present (Perkins, 1993; Olinger and Griffiths, 1993a, 1993b). The strength of these absorptions can lead to spectral distortions and nonlinearities (Culler, 1993), and could make quantitative analysis difficult or impossible in undiluted samples. Recent work, however, with a number of sample matrices including food (Downey et al., 1997; Kemsley et al., 1996; Reeves and Zapf, 1998), forage (Reeves, 1994), and soil (Janik and Skjemstand, 1995; Janik et al., 1998; Reeves et al., 2001) has demonstrated that good quantitative measurements are possible in the MIR region. These reports have demonstrated that quantitative MIRS analysis can be performed on neat (as is) samples with good accuracy.

Recent work has demonstrated good ability to establish local (within-field) NIRS and MIRS calibrations for soil C (Reeves et al., 1999; McCarty and Reeves, 2000; Reeves et al., 2001). The diversity of samples included

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Abbreviations: MIR, mid-infrared; MIRS, MIR spectroscopy; NIR, near-infrared; NIRS, NIR spectroscopy; PLS, partial least squares; RMSD, root mean squared deviation; SD, standard deviation.

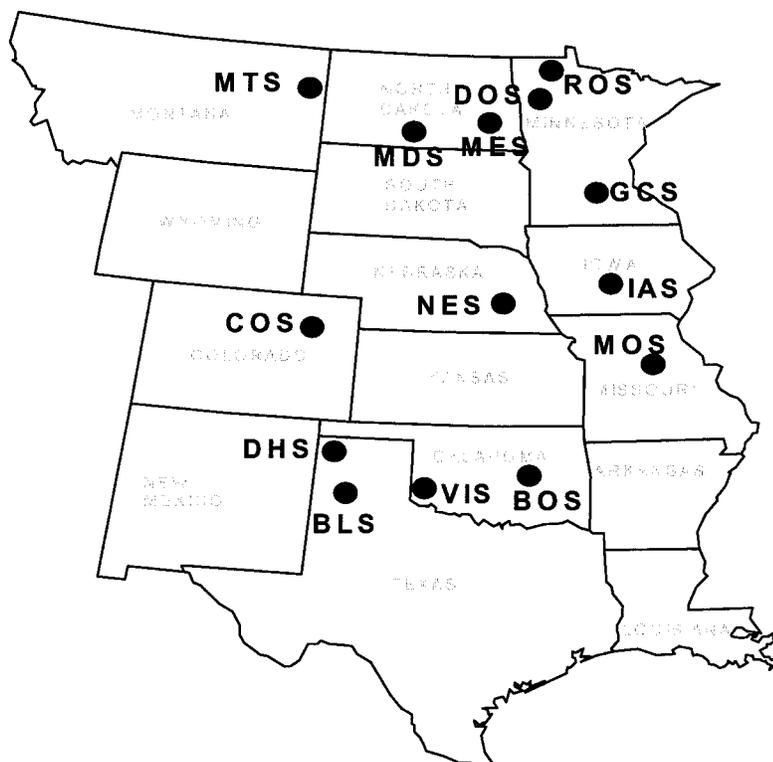


Fig. 1. Geographic location of the 14 sampling sites within the west central United States.

in these evaluations was limited to a few agricultural fields, and a question remained concerning the ability to establish broader calibrations across diverse soil types. The purpose of this study was to compare the abilities of MIRS and NIRS to measure total, organic, and inorganic C in a highly diverse set of soils and to assess feasibility of establishing regional diffuse reflectance calibrations for soil C.

MATERIALS AND METHODS

Soil Collection and Conventional Analyses

The 273 samples used in this study were soil profile samples collected as described by Follett et al. (2001) from 14 geographically diverse locations in the central United States (Fig. 1). Soil temperature regimes ranged from thermic to frigid and soil moisture regimes from udic to aridic. From each location, the soil samples were collected from adjacent parcels of land under crop production, native vegetation (never cultivated), and conservation reserve program (CRP) management. The soils were sampled to a depth of 200 cm by genetic horizons with the surface layer sampled at 0 to 5, 5 to 10, and 10 to ~25 cm (bottom of the Ap for cultivated soils). Before analyses, soil samples were air dried, mixed, sieved, and ground by a roller mill (180- μm mesh size). Soil C analyses were performed by dry combustion (1500°C) on a Carlo Erba C/N analyzer (Haake Buchler Instruments Inc., Saddle Brook, NJ¹). Total soil C was determined on unamended soil samples and organic soil C was determined on acidified soil samples. Inorganic soil C was determined by difference between total and organic soil C. The acidification procedure for removal

of soil carbonates involved addition of 100 mL of 0.33 M H_3PO_4 to 5 to 6 g of soil and shaking for 1 h. The procedure was repeated until the pH of the soil solution remained within 0.2 pH unit of that of the original acid solution (Follett et al., 1997; Follett and Pruessner, 2000). These acidified soil samples were oven dried at 60°C, ground to pass a 180- μm screen opening, and analyzed for C by dry combustion. Follett and Pruessner (2000) reported that acidification removed soil inorganic C (carbonates), but little or no organic C. However, they did caution that for some soils, acidification may remove neutral sugars and possibly other soluble organic compounds and the significance of this influence needs further investigation.

Infrared Spectroscopy

Samples were scanned in the MIR from 4000 to 400 cm^{-1} (2500–25 000 nm) at 4 cm^{-1} resolution with 64 coadded scans per spectra, on a DigiLab FTS-60 Fourier transform spectrometer (Bio-Rad, Randolph, MA) equipped with standard DRIFT optics under purge and with a custom fabricated sample transport which allowed a 50 by 2 mm^2 sample to be scanned (Reeves, 1996). Samples of ground soil were placed in the sample cell without sample dilution and no precautions were used to avoid specular reflection. Log-transformed reflectance data was used in analysis. Near infrared spectra were obtained using a NIRSystems model 6500 scanning monochromator (Foss-NIRSystems, Silver Spring, MD). Samples were scanned from 1100 to 2498 nm (PbS detector) using a rotating cup. Data were collected every 2 nm (700 data points per spectra) at a resolution of 10 nm.

Statistical Analysis

Descriptive statistics on soil properties were performed using SAS data analysis software (SAS, 1988), and analyses of NIRS and MIRS spectral were performed by Partial least

¹ Trade and company names are included for the benefit of the reader and do not imply endorsement or preferential treatment of the product by the authors or the USDA.

Table 1. Location, soil series, texture, and classification of soils studied.

Location	Map symbol†	Soil series	Texture	Taxonomic classification
Akron, CO	COS	Weld	silt loam	Fine-loamy, smectitic, mesic Aridic Argiustolls
Indianola, IA	IAS	Macksburg	silty clay loam	Fine, smectitic, mesic, Aquic Argiudolls
Dorothy, MN	DOS	Radium	loamy sand	Sandy, mixed, frigid, Oxyaquic Hapludolls
Glencoe, MN	GCS	Nicollet	clay loam	Fine-loamy, mixed, superactive, mesic Aquic Hapludolls
Roseau, MN	ROS	Percy	loam	Coarse-loamy, mixed, superactive, frigid Typic Calciaquolls
Columbia, MO	MOS	Mexico	silt loam	Fine, smectitic, mesic Aeric Vertic Epiaqualls
Sidney, MT	MTS	Bryant	loam	Fine-silty, mixed, superactive, frigid Typic Haplustolls
Lincoln, NE	NES	Crete	silt loam	Fine, smectitic, mesic Pachic Argiustolls
Mandan, ND	MDS	Farnuf	loam	Fine-loamy, mixed superactive, frigid Typic Argiustolls
Medina, ND	MES	Barnes	loam	Fine-loamy, mixed, superactive, frigid Calcic Hapludolls
Boley, OK	BOS	Stephenville	loamy fine sand	Fine-loamy, siliceous, active, thermic Ultic Haplustalls
Vinson, OK	VIS	Madge	loam	Fine-loamy, mixed, superactive, thermic Typic Argiustolls
Bushland, TX	BLS	Pullman	clay loam	Fine, mixed, superactive, thermic Torreritic Paleustolls
Dalhart, TX	DHS	Dallam	fine sandy loam	Fine-loamy, mixed, mesic Aridic Paleustalls

† See Fig. 1.

squares regression (PLS) using Grams/386 PLSPlus V2.1G (Galactic Industries Corp., Salem, NH). Efforts using a variety of data subsets, spectral data point averaging, derivatives (first and second), and other data pretreatments (mean centering, variance scaling, multiplicative scatter correction, and baseline correction) were carried out to determine the best data pretreatment for each assay. In all cases, the number of PLS factors used in the calibration was determined by the Prediction residual error sum of squares (PRESS) *F*-statistic from the one-out cross validation procedure. Once the optimal number of PLS factors was determined, a final calibration was developed. This optimum ranged between 16 to 19 factors for all soil C calibrations reported here.

Chemometrics

Chemometric analysis (Massart et al., 1998) involves use of numeric factor analysis such as PLS regression to extract information from spectral data that relates to a property measured within a population of samples with a given domain of properties (i.e., the extent of sample diversity). This population can constitute a calibration set and the resulting calibration model can be used to estimate the modeled property in new samples with properties falling within the property domain of the calibration set. The goodness of calibration for the property of interest can depend on the degree to which this property can be modeled from spectral information of samples within the property domain of the calibration set. The robustness of calibration can depend on the extent of the property domain for the calibration set. A more inclusive property domain for the calibration set may result in a greater ability to characterize samples with greater diversity, but may also degrade the ability to model the property of interest from spectral data. Spectral similarity between samples and the calibration set can provide an indication that the sample falls within the property domain of the calibration set. For example, samples with spectral data that do not properly fit the calibra-

tion model are classified as spectral outliers which may be indicative of samples outside the property domain for the calibration set or may be indicative of the quality of information used to create the model with the spectral region.

RESULTS AND DISCUSSION

The soils used in this study were collected from a large geographic region of the west central USA. Taxonomic classification of collected soils is provided in Table 1. The extensive range in content of total, organic, and inorganic C for these samples provided a good test of the influence of soil diversity on the ability of NIRS and MIRS to quantify soil C (Table 2).

Table 2. Summary statistics for properties of soils analyzed (*n* = 237).

Property	Mean	Range	Std dev.
Total C, † g kg ⁻¹	18.3	0.98–104	15.5
Organic C, ‡ g kg ⁻¹	12.1	0.23–98.0	13.2
Inorganic C, § g kg ⁻¹	6.2	0.00–65.4	10.8
pH CaCl ₂ ¶	6.5	4.10–8.80	1.1
pH H ₂ O #	7.1	4.30–8.80	1.1

† Dry combustion of untreated soil.

‡ Dry combustion of acidified soil.

§ Determined by difference between untreated and acidified soil.

¶ 1:1 suspension with 0.01 M CaCl₂.

1:1 suspension with deionized water.

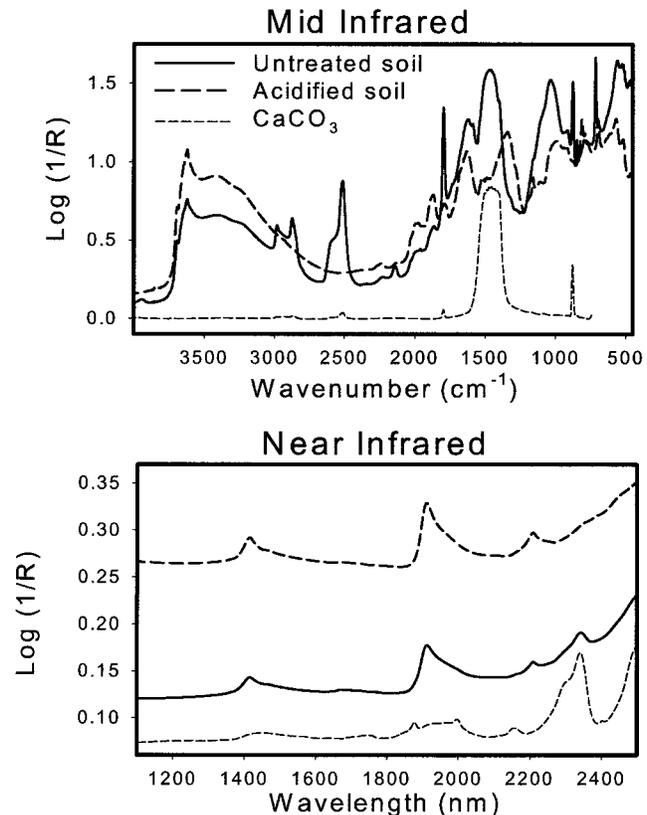


Fig. 2. Comparison of mid-infrared and near-infrared spectra of a highly calcareous soil before and after treatment with acid for removal of carbonates. The carbonate (i.e., CaCO₃) spectrum in each spectral region is included for additional comparison.

A comparison of the spectra from MIRS and NIRS for a calcareous soil shows obvious differences in the character of spectral data in the NIR and MIR regions (Fig. 2). Strong and well defined absorption features from fundamental molecular vibration modes dominate the MIR region, whereas comparatively muted and diffuse absorption features from vibration overtones and combination bands fall within the NIR region. The influence of acidifying the calcareous soil on the spectra is also shown with comparison to the spectra for CaCO_3 , demonstrating the strong influence of CO_3 on both the MIR and NIR spectra of the untreated soil. Acid treatment eliminated the absorption features correlated to the CaCO_3 spectra, providing confirmation that these features in the soil spectra were because of CO_3 absorption bands. Clearly, CO_3 has a prominent influence on MIR and NIR spectra of calcareous soils samples.

A comparison of the ability of MIRS and NIRS to measure total and inorganic soil C (Fig. 3) shows that the MIRS calibrations performed significantly better than those of NIRS. The Residual mean squared deviation (RMSD) for MIRS was essentially half that for

NIRS. The MIRS calibration for CO_3 provided a remarkably good fit ($R^2 = 0.99$) to the data considering the diversity of soil samples analyzed. Comparison of MIRS and NIRS for measurement of organic C also showed that MIRS outperformed NIRS (Fig. 4). The use of acidified samples provided better calibration for organic C in the MIR region, but degraded the ability to calibrate in the NIR region. For all calibrations, a number of spectral outliers were identified in the analysis of the NIR data which were not identified in the MIR data. This further indicates that NIRS is less robust than MIRS when developing calibrations for widely diverse soil samples.

Random exclusion of one third of the samples from the calibration set to provide an independent validation set (Table 3) showed that the validation set was predicted quite well by the calibration set, with slight introduction of bias. An independent validation set composed solely of samples from the Nebraska sampling site (Table 4) showed significantly higher bias in predictions than with the random exclusion set. Previous work demonstrated that inclusion of just a few samples from

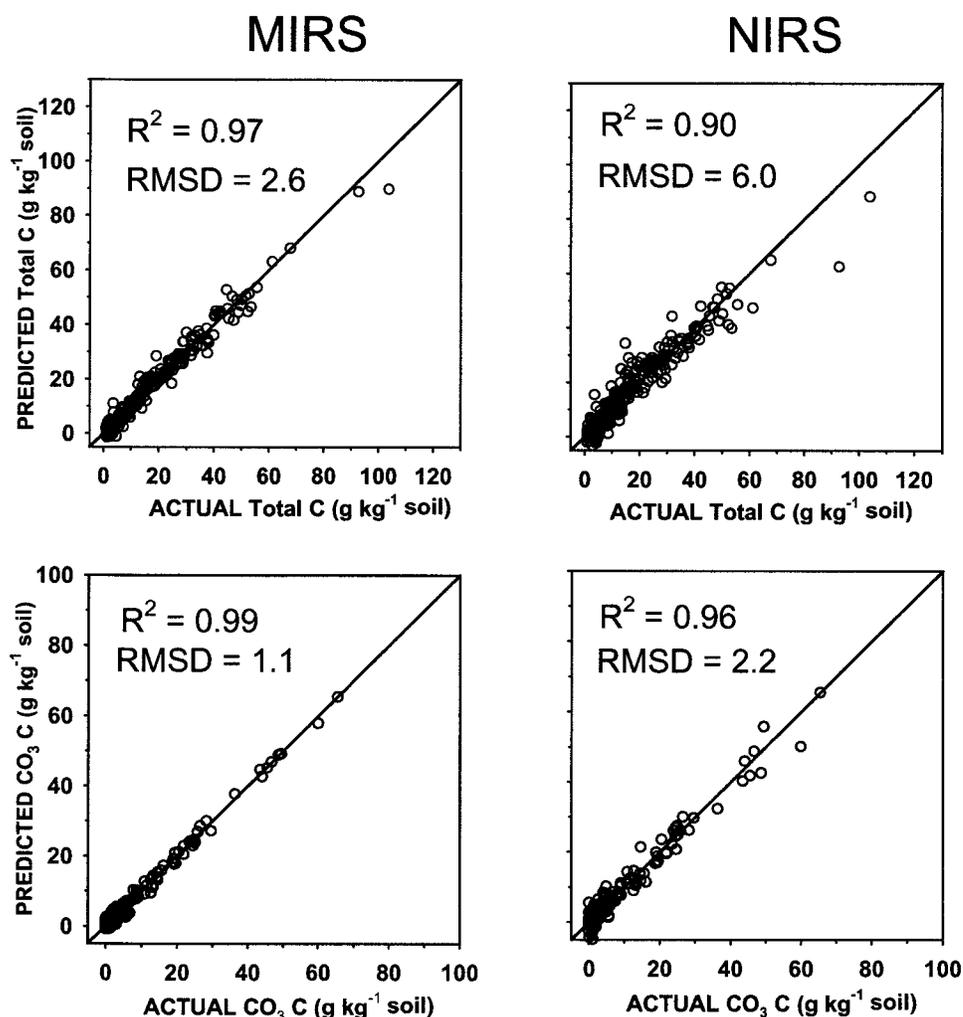


Fig. 3. Comparison of calibration for near-infrared and mid-infrared spectroscopy based on total and inorganic soil C measured by dry combustion (actual). Partial least squares (PLS) regression analysis for total C used 17 factors for mid-infrared spectroscopy (MIRS) and 16 factors for near-infrared spectroscopy (NIRS), and analysis for inorganic C used 16 factors for MIRS and 19 factors for NIRS. Residual Mean Squared Deviation is represented by RMSD.

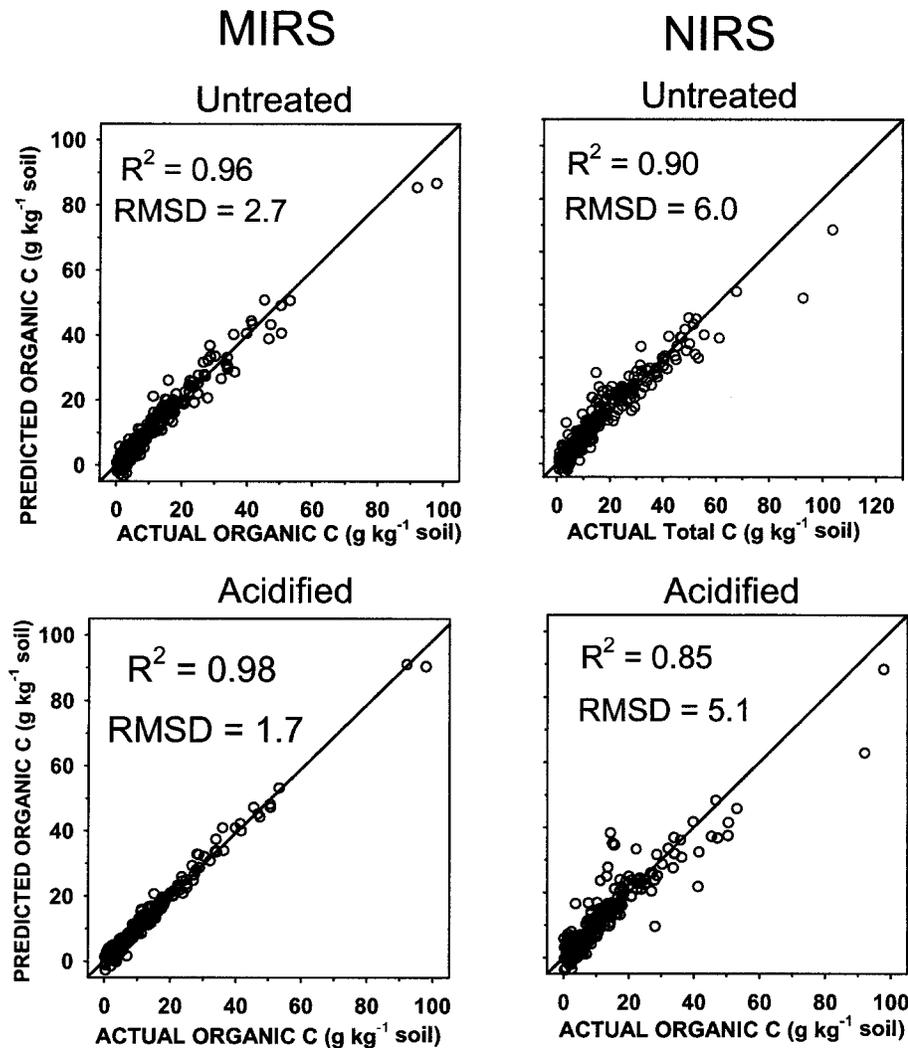


Fig. 4. Comparison of calibrations near- and mid-infrared spectroscopy based on organic C in untreated and acidified soils measured by dry combustion (actual). Partial Least Squares (PLS) regression analysis for organic C used 17 factors for mid-infrared spectroscopy (MIRS) and 18 factors for NIRS, and analysis for organic C (acid) used 19 factors for MIRS and 17 factors for NIRS. Residual Mean Squared Deviation is represented by RMSD.

a new sampling location into the calibration set can readily correct for similar location bias (McCarty and Reeves, 2000).

One of the concerns with use of MIR for quantitative analysis of neat samples has been the very strong absorption features that lead to nonlinearities and spectral distortions (Culler, 1993). Recent work has demonstrated, however, that sample dilution is not necessary with use of MIRS in conjunction with chemometric analysis of soil (Nguyen et al., 1991; Janik et al., 1998; Reeves et al., 2001). This may result partly from the fact that

with complex sample matrices such as soil, individual components often make up only a small portion of the total sample, and, therefore, the component being measured is effectively diluted by the sample as a whole. It is evident from MIR spectra of calcareous soil, however, that the CO₃ absorptions are very prominent features of the spectra. This increases the potential for associated nonlinearities and distortions and could potentially limit utility of chemometric analysis. We demonstrate, how-

Table 3. Independent validation set of 60 soil samples with the remaining samples ($n = 177$) used to develop a calibration.

Assay	MIR			NIR		
	R^2	RMSD†	Bias	R^2	RMSD†	Bias
Total C	0.95	3.4	0.4	0.86	5.4	-0.3
Inorganic C	0.98	1.2	0.3	0.87	3.1	-0.1
Organic C	0.94	3.2	0.01	0.82	5.5	0.1
Organic C (acid)	0.97	2.4	0.5	0.80	5.8	-1.1

† Residual mean squared deviation.

Table 4. Independent validation set of soil samples collected at the Nebraska location ($n = 16$) with the remaining samples ($n = 257$) used to develop a calibration.

Assay	MIR†			NIR‡		
	R^2	RMSD§	Bias	R^2	RMSD†	Bias
Total C	0.99	7.6	3.9	0.96	10.1	-5.8
Inorganic C	0.82	0.7	-0.3	0.42	4.4	-4.2
Organic C	0.98	6.0	-2.7	0.98	7.9	-2.8
Organic C (acid)	0.98	4.8	-1.8	0.98	7.1	-2.0

† Mid infrared.

‡ Near-infrared.

§ Residual mean squared deviation.

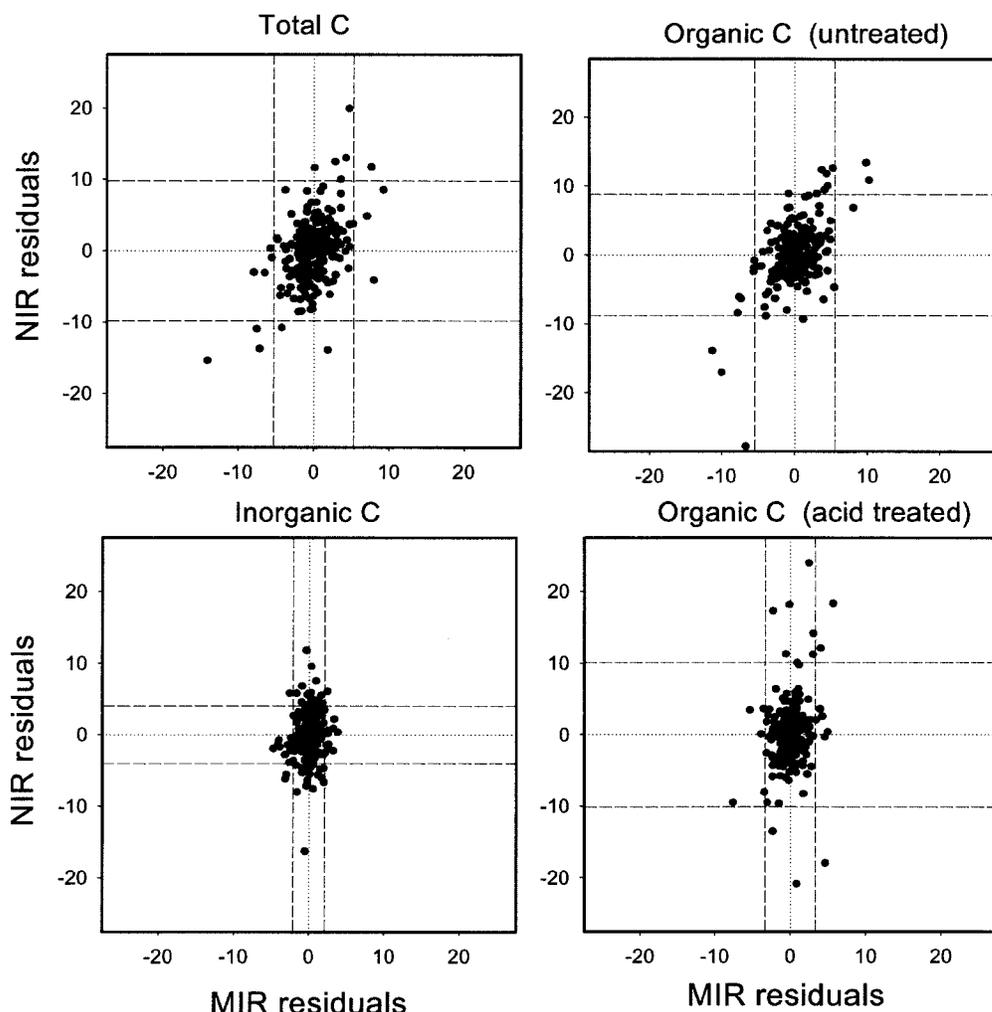


Fig. 5. Residual error distributions for mid-infrared spectroscopy (MIRS) and near-infrared spectroscopy (NIRS) calibrations. Dashed lines indicate two standard deviations (2σ).

ever, that very good calibrations can be obtained for soil CO_3 . This may result from sufficient dilution of CO_3 by other soil components and the ability of PLS analysis to handle nonlinear influences within spectral data.

We found with MIRS analysis that determination of organic C is degraded by soil CO_3 and that with acid treatment of the samples a significantly better calibration can be obtained. The cause of this degradation is not known. It is possible that the strong CO_3 signal masks spectral features important in determination of organic soil C. In the MIRS analysis of soil samples without acid treatment, a large portion of the spectral variance for that is associated with the CO_3 component of the soil. It is possible that the large pool of spectral variance associated with soil CO_3 degrades the ability to obtain information from the remaining variance associated with the soil organic C component.

Comparison of the distribution of PLS error residuals for MIRS and NIRS calibrations (Fig. 5) show that corresponding untreated soil samples tended to be poorly predicted for total C and organic C in both spectral regions. Acid treatment of soil diminished this trend for organic C estimation and resulted in a tighter residual

error distribution for MIRS, but a broader distribution for NIRS. It is noteworthy that the spread in residuals associated with measurement of total soil C (standard deviation, $\text{SD} = 2.6$) was essentially equal to the spread in residuals associated with measurement of organic C in the untreated soils ($\text{SD} = 2.7$). This indicates that most of the error in estimating total soil C in soil resulted from estimation errors of the organic C component. In fact, it may well be that a substantial portion of the residuals for estimating inorganic soil C was because of limitations in determinations based on difference between total and organic soil C. A more accurate chemical analysis for inorganic soil C could very likely cause significant improvement in MIRS calibrations for this constituent.

With the wide extent of soil properties covered by the soil samples used in this study, it is uncertain that the number of samples used (273) was sufficient to provide optimal resolution of the spectral variance needed for best estimates of soil C. It may well be that larger calibration sets are needed to cover the diversity of soil properties found over the large geographic region that was sampled. Even with this limitation, our study dem-

onstrates that useful regional calibrations for soil C can be developed using MIRS analysis and to lesser extent using NIRS analysis.

CONCLUSIONS

Accurate assessment spatial distribution and inventory of below ground C within landscapes is data intensive, and resource-intensive methods of analysis such as dry combustion are not well suited for generating the necessary soil C data. Use of spectral analysis for measurement of soil C has advantage in that the method is nondestructive, consumes no reagents, and highly adaptable to automated and in situ measurements. In addition, our work demonstrates that spectral analysis permits simultaneous measurement of organic and inorganic soil C which simplifies analysis relative to the traditional chemical methods. Development of instrumentation specific for soil analysis holds promise for rapid and automated means of C measurement. For example, we have subsequently used an infrared spectrometer fitted with a 60-sample autosampler which demonstrated ability to analyze samples within a time scale of 1 min. Most of the work on spectral analysis for quantifying soil properties has been focused on use of NIRS, but our comparison of NIRS and MIRS provides strong evidence that MIR spectral contain better information related to soil C. These results indicate that greater effort should be given to developing MIRS instrumentation for environmental sampling and approaches to extract information contained in MIR spectra on soil composition.

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