

Prediction of carbon mineralization rates from different soil physical fractions using diffuse reflectance spectroscopy

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Abstract

Soil carbon (C) mineralization rate is a key indicator of soil functional capacity but it is time consuming to measure using conventional laboratory incubation methods. Recent studies have demonstrated the ability of visible-near infrared spectroscopy (NIRS) for rapid non-destructive determination of soil organic carbon (SOC) and nitrogen (N) concentration. We investigated whether NIRS (350–2500 nm) can predict C mineralization rates in physically fractionated soil aggregates (bulk soil and 6 size fractions, $n = 108$) and free organic matter (2 size fractions, $n = 27$) in aerobically incubated samples from a clayey soil (Ferralsol) and a sandy soil (Arenosol). Incubation reference values were calibrated to first derivative reflectance spectra using partial least-squares regression. Prediction accuracy was assessed by comparing laboratory reference values with NIRS values predicted using full hold-out-one cross-validation. Cross-validated prediction for C respired (500 days) in soil aggregate fractions had an R^2 of 0.82 while that of C mineralized (300 days) in organic matter fractions was 0.71. Major soil aggregate fractions could be perfectly spectrally discriminated using a 50% random holdout validation sample. NIRS is a promising technique for rapid characterization of potential C mineralization in soils and aggregate fractions. Further work should test the robustness of NIRS prediction of mineralization rates of aggregate fractions across a wide range of soils and spectral mixture models for predicting mass fractions of aggregate size classes.

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1. Introduction

In order to determine the rate and/or extent of carbon (C) storage and protection in soils an obvious requirement is the need to measure the soil C content. Methods such as dry combustion and chemical-based techniques are time consuming, expensive and not adaptable to in situ determinations. Among techniques that can be utilized to enable faster and economically favourable assessment of soil C and its turnover, diffuse reflectance spectroscopy recently has been widely tested for the rapid non-destructive characterization of soil organic carbon (SOC) in soils (Morra et al., 1991; Ben-Dor and Banin, 1995; Couillard et al., 1997; Shepherd and Walsh, 2002; Reeves et al., 2002).

Spectral signatures of materials are defined by their reflectance or absorbance, as a function of wavelength in the electromagnetic spectrum. Fundamental features related to various components of soil organic matter occur at energy levels that allow molecules to rise to higher vibrational states. These features occur in the mid- to thermal-infrared range (2500–25,000 nm), but their overtones occur in the near- (700–1000 nm) and short-wave infrared (1000–2500 nm) regions (Shepherd and Walsh, 2002). Soil organic matter also influences soil spectra in the visible range (Ben-Dor et al., 1999), which is now included in commercially available instruments (Fystro, 2002). Near- and mid-infrared spectroscopic determinations require the development of calibrations that relate the analyte in question, i.e. C content in soils, to the spectral information using multivariate statistical procedures such as principal components regression (PCR) and partial least-squares regression (PLSR) among others (Workman, 1992).

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In addition to the use of near-infrared spectroscopy for determining soil organic matter content and total nitrogen (N) contents, its potential has been demonstrated for estimating leaf litter chemistry, litter mass remaining during decomposition stages (Gillion et al., 1993), organic matter quality induced variation in rates of respiration (Palmborg and Nordgren, 1993; Joffre et al., 2001), and organic resource quality (Shepherd et al., 2003). Recently, Fystro (2002) has demonstrated the use of spectral spectroscopy in predicting C and N mineralization in grassland soils. This study aimed to test the potential of visible-near-infrared reflectance to predict C mineralization in physically separated water stable aggregates (WSA, 20–2000 μm), microaggregates (MI, <20 μm) and organic matter fractions.

2. Materials and methods

2.1. Soils and land uses

Soils were collected from soil management experiments in agricultural fields conducted at two sites in western Kenya, in Luero (a Ferralsol) and Teso (an Arenosol), selected to provide variation in soil fraction mineralization rates due to both site (principally particle size distribution) and management effects. The sites varied widely in sand content and SOC in the top 5 cm layer (Table 1). Soil samples for incubation were taken in August 2001 from plots under continuous maize cropping (two maize crops per year, for at least 5 years) and 5 months after harvesting an 18 month-old fallow of *Tephrosia candida* on a plot that had previously been under continuous maize cropping (with returning of leaves, pods and twigs to the soil). The *Tephrosia* had been planted as an improved fallow for soil fertility recapitalization (Mutuo, 2004). Each of the two cropping treatments was replicated four times in a randomized block design, giving eight sampled plots at each of the two sites.

2.2. Fractionation of WSA, MI and free organic matter (fOM) fractions

The approach to characterize WSA was based on a multiple-wet sieve technique (Gregorich et al., 1989; Feller et al., 1996). The method takes into account the energy input level, and consists of whole aggregate size analysis from the macroaggregates (>500 μm) to the water-dispersible clay (0–5 μm) fraction. Aggregates separated after re-wetting the soil and shaking for 1 h in water (method of Gregorich et al., 1989; Feller et al., 1996) are thought to offer increased protection due to absence of external pores, and hence poor access by microbes to internal protected C. Samples of 50 g air-dried 2-mm sieved soil were shaken in 300 ml water for 1 h in a 50 revolutions per minute (rpm) tumbler shaker, followed by wet sieving using tap water through 500, 212, 53 and 20 μm , then separated at 5 μm by repeated gravity sedimentation. The

Table 1

Soil chemical and physical characteristics of the study sites in the top 0–5 cm depth (Mutuo, 2004)

Site	mg g ⁻¹ soil				
	SOC	TSN	Clay	Sand	Silt
Luero	18.44	1.48	420	350	230
Teso	9.8	0.59	180	760	60

SOC = Soil organic carbon and TSN = Total soil nitrogen.

fractionation procedure used is represented in a diagram in Fig. 1. Fractions greater than 20 μm were separated for the aggregate and fOM components. These fractions were obtained by transferring the respective size classes into a 3–5 litre basin with some water and the fOM was obtained by slowly swirling the solids until the organic fraction floated and it was possible to separate it, while the aggregates remained at the bottom. The separated fOM and aggregates were oven-dried at 40 °C for about 72 h and weighed. The resultant moisture content of fractions was about 2%.

2.3. Incubation procedure

Samples of whole soil, aggregates and fOM fractions were incubated in triplicate in 500 mL glass jars. The weights of incubated samples varied from 0.5 g for fOM samples to 10 g for soil and aggregates greater than 53 μm , while 5 g samples were used for aggregates less than 53 μm . During the incubation, aggregates less than 53 μm and fOM were mixed with 5 g of acid-washed sand to improve aeration. Samples were wetted to field capacity. Small glass bottles were fitted within the jars containing 10 ml of 0.25 M NaOH to trap the CO₂ evolved. Jars were sealed and stored in a dark room at 27 °C. Jars were opened regularly and the NaOH changed for up to 500 (for soils and aggregate fractions) and 300 days (for fOM fractions). C evolution was determined by pipetting 5 ml of the C-containing NaOH, and autotitrating with 0.15 M HCl after precipitation of carbonates with 8 mL of 3 M BaCl₂ (Haber, 1958).

2.4. Measurement of soil properties

Total C and N in samples were determined by dry combustion using a Roboprep automatic C/N analyzer (Europa Scientific, Crewe, UK) with a biological sample converter coupled to a Europa Scientific 20–20 stable isotope mass spectrometer.

Diffuse reflectance of samples was measured on air-dried soil (2.8 ± 0.2% moisture content) and oven-dried (at 40 °C for about 72 h) physically separated aggregates and fOM fractions (2.0 ± 0.4% moisture content), the same fractions incubated to measure C mineralization. Reflectance spectra were recorded for each sample at 1-nm intervals from 350

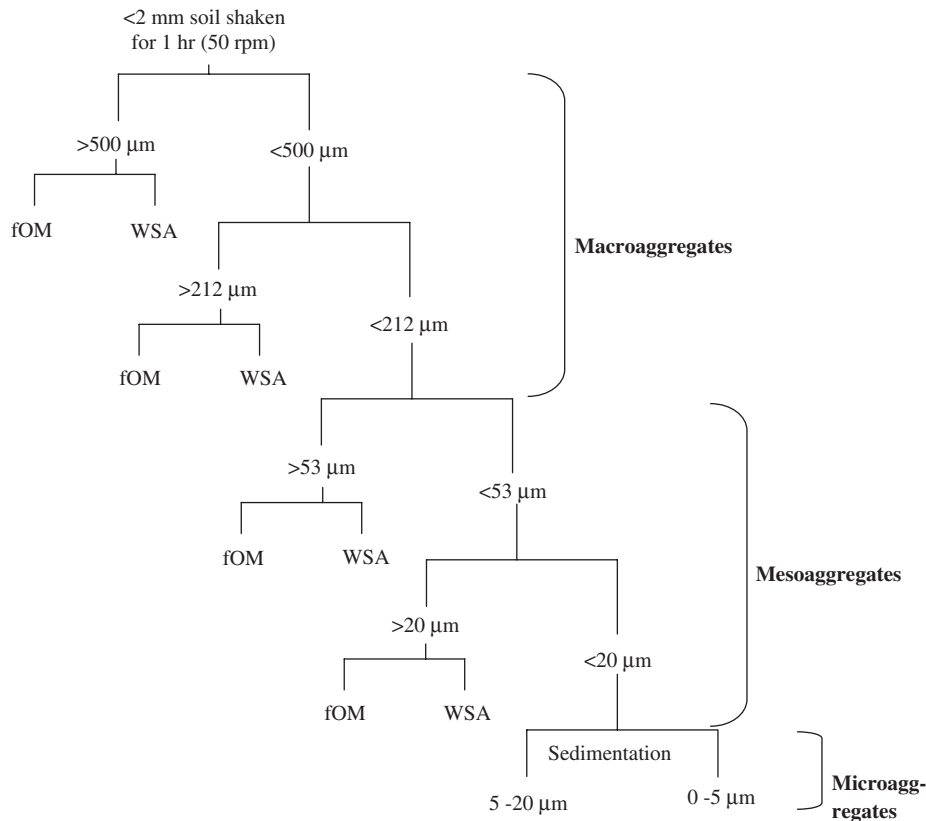


Fig. 1. Procedure used for physical fractionation of soil into water stable aggregates and microaggregates. Separation of fOM from WSA was done using floatation in water, while separation of fine silt and clay-sized microaggregates was achieved by sedimentation.

to 2500 nm using a FieldSpec™ FR spectroradiometer (Analytical Spectral Devices, Boulder, CO). The density of data was reduced by selecting every 10th-nanometer value to ease data handling and match the data more closely to the spectral resolution of the instrument (3–10 nm). Enough sample was placed into 7.4 cm diam. Duran glass Petri dishes to give a sample thickness of about 1 cm. For small samples of organic matter fractions, the sample was covered with an optically black plate to eliminate any light interference effects. The samples were scanned through the bottom of the Petri dishes using a high-intensity source probe (Analytical Spectral Devices, Boulder, CO). The probe illuminates the sample (4.5 W halogen lamp giving a correlated colour temperature of 3000 K; WelchAllyn, Skaneateles Falls, NY) and collects the reflected light from a 3.5 cm diam. Sapphire window through a fibre-optic cable (Shepherd et al., 2003).

To sample within-dish variation, reflectance spectra were recorded at two positions, successively rotating the sample dish through 90° between readings. The average of 25 spectra (the manufacturer's default value) was recorded at each position to minimize instrument noise. Before reading each sample, 10 white reflectance spectra were recorded using a calibrated spectralon (Labsphere, Sutton, NH) placed in a glass Petri dish. Reflectance readings for each wavelength band were expressed relative to the average of the white reference readings.

2.5. Statistical analysis

Total C and N, and C mineralization were calibrated to relative reflectance using the 16 soil samples, 87 aggregate fractions and 28 fOM fractions from the two sites. Nine aggregate and four fOM fractions were quite small to enable reliable near-infrared spectroscopy (NIRS) readings. Spectra were transformed with first-derivative processing (differentiation with second-order polynomial smoothing with a window width of 20 nm) using a Savitzky-Golay filter, as described by Fearn (2000). Derivative transformation is known to minimize variation among samples caused by variation in grinding and optical setup (Martens and Naes, 1989). Wavebands in the regions of low signal/noise ratio or displaying noise due to splicing between the individual spectrometers (Analytical Spectral Devices, 1997) were omitted, leaving 203 wavebands for analysis. The omitted bands were 350–420, 970–1010, and 2500 nm.

Partial least-squares regression was used to calibrate the target variables to soil reflectance using The Unscrambler® software (CAMO, Oslo, Norway). Hold-out-one full cross-validation with jack-knifing (Martens and Martens, 2000) was used to evaluate the stability of the calibrations and to eliminate unreliable (non-significant) wavebands in the calibrations. The target variables were transformed to give a normal distribution where necessary but prediction

Table 2

Chemical and C mineralization data of soils and aggregates ($n = 103$) and free organic matter fractions ($n = 28$)

	Soils and aggregates				Free organic matter	
	Luero		Teso		(Both sites)	
	Range	Mean	Range	Mean	Range	Mean
C (mg g^{-1})	16.15–29.62	22.15	1.07–48.04	14.67	87.8–307.7	182.0
N (mg g^{-1})	1.57–3.18	2.28	0.06–4.89	1.28	5.7–14.7	11.8
C:N ratio	7.14–12.44	9.87	9.31–18.73	13.56	11.4–23.0	15.3

performance was evaluated on back-transformed data. No outliers were omitted from the analysis. Prediction performance was assessed using hold-out-one cross-validation, plotting actual versus predicted values, and reporting the R^2 , root mean-square error (RMSE) and bias.

Ability to discriminate soil aggregate and organic matter fractions from reflectance spectra of the individual fractions soil was tested using Treenet stochastic gradient boosting (Steinberg et al., 2002) as described in Shepherd et al. (2003). Aggregate and organic matter fractions were treated as the target class variable and calibrated to the first derivative reflectance values of the individual fractions using random hold-out validation stratified by fraction class.

3. Results and discussion

3.1. Chemical data and C mineralization

C and N contents in soils and aggregates ranged from 1.1 to 48 mg g^{-1} for all soils, but the range was wider in the sandy soil (Teso) than in the clayey soil (Luero) (Table 2). The observed lower concentrations of C and N were those of macroaggregates in the sandy soil because of their high proportion of sand (about 85%) (Mutuo, 2004). The highest C and N-values were from the microaggregates of both soils. C:N ratios were higher (averaging about 13.6) in soils and aggregates of the Teso site compared to those from Luero (averaging about 9.9) (Table 2) probably because of the existence of most of the soil C in the form of fOM in the sandy soil, and hence the higher C:N ratio. Both the cumulative and rate of percent C evolved during 500 days of incubation from soils and aggregates of the sandy soil were about twice those from the Ferralsol (Table 2). Lower C mineralization from soils and aggregates of the clayey soil (on proportional basis) compared to those of sandy soil demonstrated the importance of clay content in C protection against microbial attack.

The proportion of C mineralized per day was greater in the larger aggregates than in smaller ones in the Arenosol, but there was no strong influence of aggregate size on respiration rates in the heavy clayey soil (Fig. 2(A)). The greater proportion of respired C from macroaggregates (500–2000 μm) of the sandy soil was attributed to the

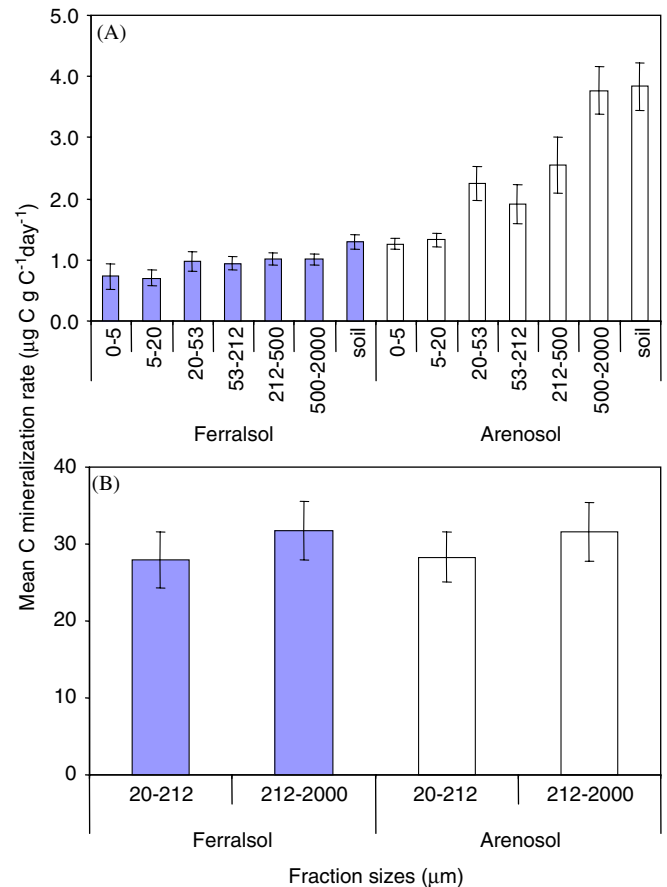


Fig. 2. Mean C mineralization rates ($\mu\text{g C g}^{-1} \text{ fraction day}^{-1}$) from soils and different aggregate size classes (A) and fOM fractions (B). Error bars are standard error of means.

poor protection of C against microbial respiration compared to smaller aggregates, which provide greater C protection.

C and N contents in fOM fractions were 182 and 12 mg g^{-1} on average, respectively (Table 2). The fOM fractions represented a more labile C pool compared to the whole soil and aggregates, because the proportion of C respired was $28\text{--}32 \mu\text{g C g}^{-1} \text{ day}^{-1}$ from the fOM fractions compared to $0.7\text{--}3.9 \mu\text{g C g}^{-1} \text{ day}^{-1}$ from soils and aggregates (Fig. 2(A) and (B)). However, the proportion of C mineralized per day from the two fOM sizes were similar (Fig. 2(B)).

In the clayey soil, the total amount of C evolved from all fractions was 2.34 mg C g^{-1} soil, while that mineralized from the whole soil was 2.46 mg C g^{-1} . The total amounts of C mineralized from fractions (1.96 mg C g^{-1}) and whole soil (1.71 mg C g^{-1}) in the sandy soil were smaller than those mineralized from the clayey soil. The apparently greater C mineralization (in absolute values) from the clayey soil as compared to the sandy soil was an expected outcome because of the larger amounts of C in the clayey soil. C mineralization from fractions and whole soils in both soil types were congruent, showing that the fractionation of soils into aggregates and fOM fractions did only to a minor extent affect the biological activity of these soils.

3.2. Spectral features

There was a distinct difference in albedo (relative reflectance over the entire spectrum) between the Ferralsol (ranging from 0.06 to 0.38) and the Arenosol (ranging from 0.15 to 0.49) (Fig. 3). The relative reflectance of soils and the aggregate fractions from each site were grouped together in Fig. 3 because they clustered together. This could be associated with the high proportion of total soil organic C contained in aggregates (90% and 70% in clayey and sandy soils, respectively), although other workers have shown that differences in soil albedo are broadly related to soil organic matter concentrations (Ben-Dor et al., 1999). The untransformed soil spectra showed prominent absorption features around 1400, 1900, and 2200 nm (Fig. 3). These absorption features are typical in soils (Ben-Dor et al., 1999; Shepherd and Walsh, 2002), and are associated with clay minerals, for example OH features of free water at 1400 and 1900 nm, and lattice OH features at 1400 and 2200 nm (Hunt, 1982).

fOM fractions exhibited strong absorption in the region 500–850 nm than both soils (Fig. 3), which is the edge of a large absorption feature caused by lignin, centred at 280 nm (Schubert, 1965). Maximum relative reflectance of fOM was 0.49 (at 1870 nm), unlike that obtained in litter of

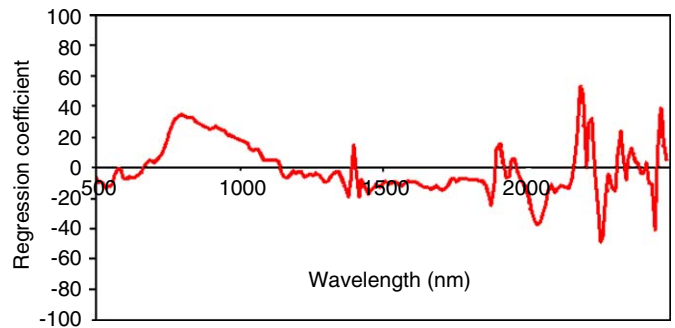


Fig. 4. Partial least-squares regression coefficients for C mineralization in soils and physically separated fractions against bulk soil reflectance in different wavebands.

0.7 (1300 nm) by Shepherd and Walsh, (2003), signifying that the association of fOM with soil mineral particles causes greater absorption overall. Additionally, there were relatively strong absorption features in the NIR centred at 1470, 1940 and 2100 nm, similar to the soils, confirming the presence of soil particles due to their direct association (encrusting) with plant debris (fOM) and/or due soil contamination associated with the separation procedure. Elvidge (1990) found that lignin spectral features tended to predominate in decayed plant material, with a broad absorption between 2050 and 2140 nm.

C mineralization from soils and physically separated aggregates and fOM fractions displayed large absolute regression coefficients with derivative spectra at 800, 2030, 2180, 2200, 2250, 2440, 2460 nm (Fig. 4). These may reflect differences in the amount of organic matter protection between soil types and fractions due to effects of iron oxides around 800 nm, and clay mineralogy, especially lattice OH effects around 2200 nm (Hunt, 1982).

3.3. Prediction of chemical data and respiration by NIRS

In soils and aggregates, cross-validated prediction of total C contents ($R^2 = 0.84$) and total N contents ($R^2 = 0.87$) were good overall, but there was much scatter within the Ferralsol (Fig. 5(A)). In fOM fractions (20 μm –2 mm in size) predictions of total C ($R^2 = 0.97$) and total N ($R^2 = 0.91$) contents were good (Fig. 5(B)), while that of C:N ratio was poor ($R^2 = 0.37$, not presented). Previous studies have shown that soil C and N contents can be predicted well by spectroscopy (Ben-Dor and Banin, 1995; Couillard et al., 1997; Shepherd and Walsh, 2002; Reeves et al., 2002; Fystro, 2002; Shepherd et al., 2003). Results from this study further showed that NIRS is a promising tool for predicting C and N contents of physically fractionated aggregates and fOM fractions in different soils from the bulk soil spectrum.

Spectral calibration for organic C and total N was better for the fOM fractions than for soils and aggregates (Fig. 5) probably because of the interference of mineral fractions (largely clay) in soil and aggregates. Soil clay minerals have very distinct spectral signatures in the short-wave infrared

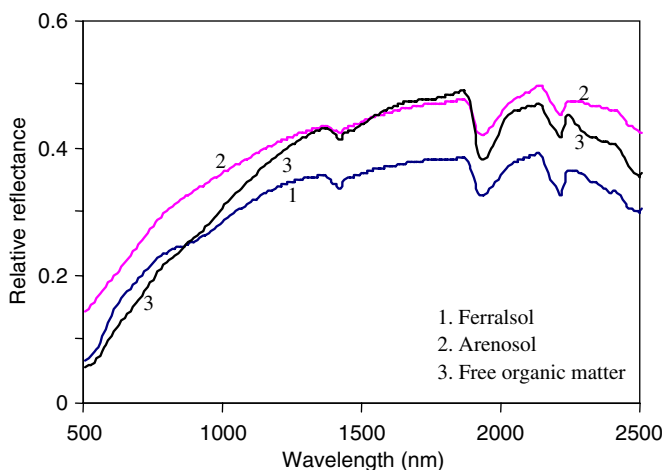


Fig. 3. Reflectance spectra for the two soil types (Ferralsol and Arenosol) and fOM fractions.

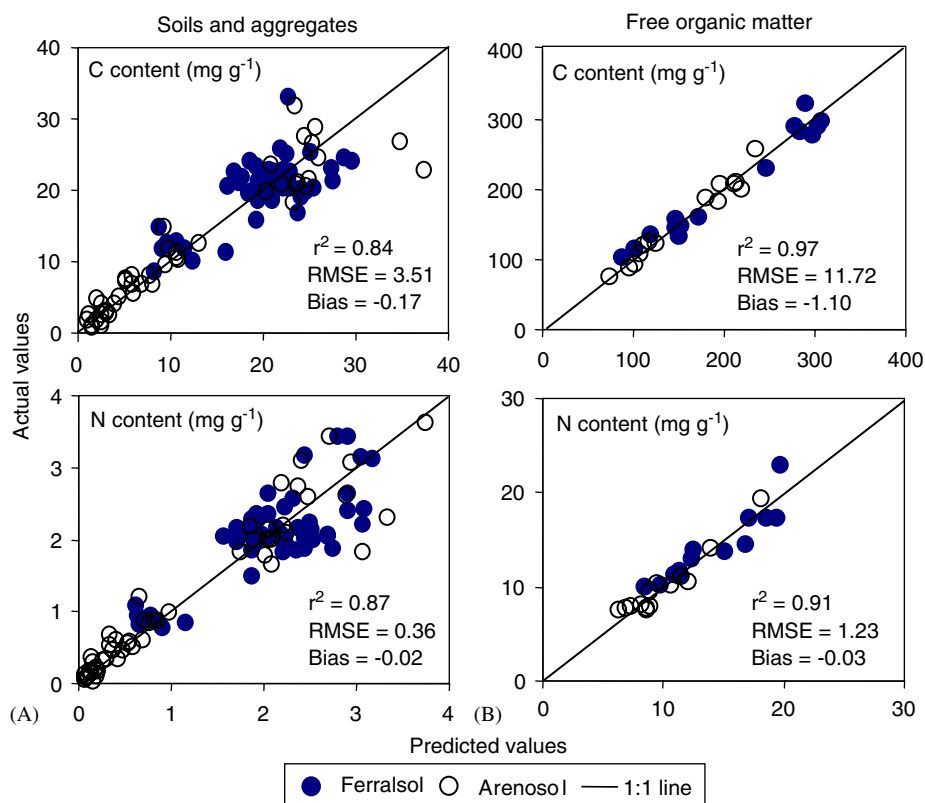


Fig. 5. Actual versus cross-validated predicted values for organic C and total N in (A) soils and aggregates ($n = 103$), and (B) fOM fractions ($n = 28$) by reflectance spectroscopy of bulk soil.

region (1000–2500 nm) because of strong absorption of overtones of SO_4^{2-} , CO_3^{2-} and OH^- (Hunt, 1982; Clark, 1999). This interference of clay minerals probably also explains why there was much scatter in C calibrations within soils and aggregates for the Ferralsol (Kaolinitic) and not for the sandy soil. If all soil fractions and whole soils were considered together, despite the big variation in C and N contents between fOM fractions and soils/aggregates, the validation R^2 were 93% for C and 94% for N. These prediction efficiencies were similar to those of individual soil and aggregates, and fOM fractions.

Good overall predictions were obtained for average C respiration in $\mu\text{g C g}^{-1} \text{ day}^{-1}$ ($R^2 = 0.77$) and mg C g C^{-1} evolved in 500 days of incubation of soils and aggregates ($R^2 = 0.82$) (Fig. 6). The R^2 for C mineralization ($\mu\text{g C g C}^{-1}$) of fOM after 300 days of incubation by NIRS was also high at 0.71 (Fig. 6). Bias was small for all variables predicted, but this is generally expected when using cross-validation. These results suggest that C mineralization and C respiration rates from physically separated soil fractions can be predicted by spectroscopy, thus expanding the applicability of the method beyond the reported success in estimating variation in rates of respiration induced by variation in organic matter quality (Palmborg and Nordgren, 1993) and C and N mineralization in grassland soils (Fystro, 2002).

Spectral reflectance signatures were able to perfectly discriminate four fraction classes (macro, meso, and micro-

fractions, and organic matter) across the two sites using a 50% randomly selected validation sample ($n = 64$). The wavebands on the shoulder of the 1900 nm and 2200 absorption features (1940 and 2250 nm) were highly diagnostic in classifying the aggregates, reflecting difference in OH vibrations in adsorbed water and lattice water, respectively. Reflectance around 2200 nm, but not 1900 nm, had high a large loading in the PLS model for C mineralization, suggesting the good prediction of C mineralization may not have been entirely due to the ability to spectrally discriminate fractions. This demonstrates the information richness in soil reflectance spectra providing ability to provide information simultaneously on different soil properties. Further work should investigate whether the ability to spectrally discriminate aggregate fractions can be used to develop spectral mixture models to predict mass fractions of aggregate size classes across a wide range of soils.

4. Conclusions

Soil aggregate size and soil type/texture have an important influence on protection of organic C in soils so that C mineralization rates vary widely across different soil types and soil aggregate fractions. This study showed that these effects can be calibrated to soil diffuse reflectance in the visible-near-infrared range. Good prediction of chemical and C mineralization data in soils/aggregates and fOM

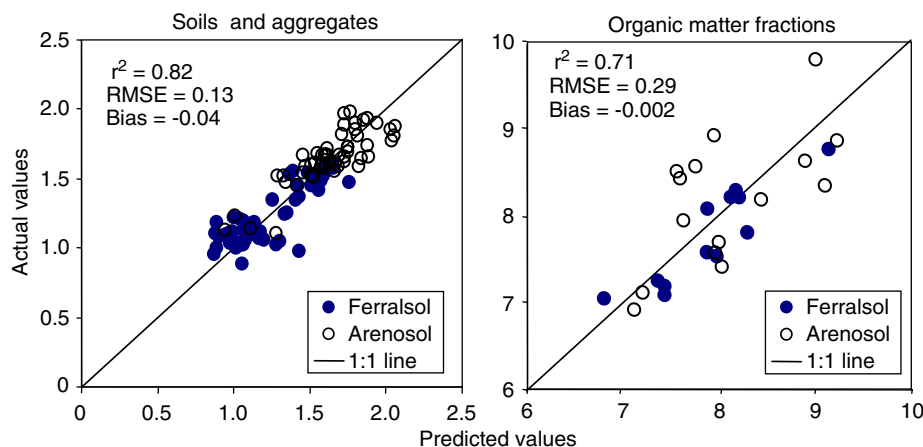


Fig. 6. Actual versus cross-validated predicted values for over-all rate of C mineralization in aggregates and soils ($n = 103$) and fOM fractions ($n = 28$) ($\mu\text{g C g}^{-1}$) by reflectance spectroscopy of bulk soil.

fractions using NIRS suggests that the method potentially captures the same information from the different soil fractions. Thus, NIRS may be useful in evaluating C storage potential in soils. However, evaluations of potential C mineralization of soil fractions from sandy soils need further investigation. For wider application, we would recommend calibrations of chemical data and C mineralization across a wide range of soils in a target area using the spectral library approach (Shepherd and Walsh, 2002). NIRS in such applications may reduce tedious laboratory analysis and permit denser sampling strategies.

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