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Evaluation of methods for measuring soil organic carbon in West African soils

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Increased interest in implementing projects in the Sahel region of Africa for sequestration of atmospheric carbon dioxide in soil organic matter has intensified the need for methods that accurately measure soil C but which are also suitable for use by most resource limited soil analysis laboratories in the region. The challenge is amplified by the low carbon content of the degraded soils found on agricultural lands in the region (often less than 5 g kg⁻¹ soil). Various indirect methods have been recommended for estimating soil C which can be readily implemented in a limited resource environment but they require more evaluation in terms of accuracy. This study compares soil carbon results obtained by direct elemental analysis using dry combustion to estimates obtained by indirect methods such as dichromate oxidation, mass loss-on-ignition (LOI), and diffused reflectance infrared spectroscopy that has advantages in being simple, inexpensive, and/or rapid. The results showed that both dichromate oxidation and loss-on-ignition had substantial limitations when used to analyze the low carbon soils within the region. Diffused reflectance spectroscopy using either the near- or mid-infrared spectral regions performed well for predicting soil organic carbon as well as sand, silt and clay content. These results indicate that once calibrated, infrared spectroscopy holds great promise for quantifying soil properties in the Sahel region and that regional laboratories could adopt the technology if instrumentation were made available.

Key words: Soil organic carbon, loss on ignition, dichromate oxidation, dry combustion.

INTRODUCTION

The increasing atmospheric carbon dioxide (CO₂) content has stimulated research to assess the role of terrestrial ecosystems in the global carbon (C) cycle. The terrestrial biosphere is an important component of global C budget, but estimates of C sequestration in terrestrial ecosystems are partly constrained by the limited ability to assess changes in soil C storage. Agricultural croplands have a great potential for sequestering atmospheric C with adoption of farming practices that improve soil quality such as no-till, organic, and perennial cropping (Lal,

2004).

There is recognition that C sequestration in developing countries in sub-Saharan Africa could play a vital role in economic development (Sanchez, 2000; Antle and Uehara, 2002). In addition, food security within the African Sahel is not only adversely affected by climate change associated with decreased rainfall but also by decreased soil fertility and soil C loss (Pieri, 1992; Traoré et al., 2000; Doumbia et al., 2008). Increased C sequestration can help reverse the decline in soil fertility in the region and thereby add to food security. Approaches similar to the Clean Development Mechanism (CDM) under the Kyoto Protocol could provide a means to transmit development aid based on activities that sequester atmospheric C (Ringius, 2002). However, the

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Kyoto Protocol only included afforestation and reforestation activities within its first five year period principally due to concerns about uncertainties associated with measurement and fate of C sequestered in soils.

As part of the movement toward generating finer scale soil maps for Africa and more generally the World, there is an important initiative to establish better ground truth infrastructure for soils in sub-Saharan Africa (Sanchez et al., 2009). A significant aspect of the initiative is the development of soil measurement capability within the region. Much of the historic data for soil C content within ecosystems is based on indirect measurements such as dichromate oxidation or loss of sample mass after ignition in a muffle furnace (typically at 400°C).

Only recently, routine soil C assessments have been widely based on elemental measurements such as detection of CO₂ produced by dry combustion of a soil sample (Franks et al., 2001). Most soil analytical laboratories world wide have produced large datasets for estimating soil C based on either dichromate oxidation or mass loss on ignition.

Nelson and Sommers (1996) listed 13 published variations in dichromate oxidation methodology for estimating soil C, including that described by Walkley and Black (1934), with each variation in methodology having its own conversion factor for translating the indirect measurement to estimates of soil C. Nelson and Sommers (1996) also listed 14 published variations for ignition conditions (temperature and time) used for mass loss on ignition protocols with each having its own regression equation to convert mass loss to estimates of soil organic matter. Because of the instability of these indirect methods in the literature, the potential for analytical drift between variations in method seems great. An attempt to adjust a national soil C database for analytical drift is demonstrated by a national survey in Australia that compared dichromate oxidation results from various soil analytical laboratories throughout the continent with those from elemental analysis by dry combustion (Skjemstad et al., 2000). Such efforts to cross correlate method results can lead to increased confidence in use of these historic datasets in assessing trends in C stocks in regions of the world.

Capturing legacy datasets is also an important aspect of the global digital soil mapping initiative as recently outlined by Sanchez et al. (2009). Shifts in analytical methodology have impacts on the quality of legacy databases for soil C and could alter the degree to which we understand changes in soil C stocks impacted by land management. In developed countries, there is increasing trend towards elemental analysis of soil C. For example in the year 2000, the U.S. Natural Resources Conservation Service (NRCS) and National Soil Survey Center (NSSC) terminated soil C analyses based on dichromate oxidation because of environmental concerns about toxic waste generation (Franks et al., 2001). However, cost of analysis is still an important driver

affecting the methodologies used. For example, most soil testing laboratories in the U.S. use mass loss on ignition for routinely measuring soil organic matter (Franks et al., 2001; Palmer, 2002). A 2001 survey that compared prices charged by soil analytical laboratories in the U.S. for C analyses by loss on ignition, dichromate oxidation and dry combustion showed that loss on ignition was least expensive with relative price ratios of approximately 1:3 and 1:4 for dichromate oxidation and dry combustion respectively (Palmer, 2002).

A rapidly developing analytical technology based on chemometric analysis of near infrared (NIR) or mid infrared (MidIR) spectra of soil shows promise to increase speed and reduce costs of soil fertility assessments (Janik et al., 1998; Reeves et al., 2001; McCarty and Reeves, 2001; 2006). McCarty et al. (2002) demonstrated that regional calibrations for soil C can be developed using these spectral ranges.

The Inter-governmental Panel on Climate Change (IPCC, 2000) concluded that an established infrastructure for measurements of soils C within developing countries would be optimal for implementation of development schemes based on soil C sequestration though such capability usually does not exist. In this report we compare various options for measurement of soil C within the Sahel region. Given resource limitations within the region, an objective is the identification of methods most suited for accurate measurements of C in regional soils.

MATERIALS AND METHODS

Chemical and physical analyses

Soil samples were obtained from agricultural fields within three geographic areas in the West African country of Mali (Figure 1). These soils have been also classified as Paleustalfs by Soil Taxonomy (Soil Survey Staff, 1975). The region of study is located in Sub-Saharan Africa with average annual rainfall of between 600 to 1200 mm. Soil organic C contents in the region are generally very low with sandy soils that have low clay content (Bationo and Buerkert, 2001). Soil samples were collected from fields under ridge tillage (Aménagement en Courbes de Niveau) management (Gigou et al., 2006). At each of 58 sampling locations, soils were collected at 0 - 20 cm (surface) and 20 - 40 cm (subsurface) depths for a total of 116 samples. These samples were air-dried and then crushed before storage in bulk form. Subsamples were oven dried and ground by roller mill for 24 h before analyses for C and N by dry combustion (Leco CNS 2000, St. Joseph, MI). Particle size analyses were performed by sedimentation and use of a hydrometer (Gee and Bauder, 1986).

Dichromate oxidation

The analyses were performed at the Soil and Plant Laboratory in the Institut d'Economie Rurale, Bamako Mali using a dichromate oxidation procedure described by Anne (1945). Soil samples (60-mesh) that contain approximately 30 mg organic C were placed into a 150-mL flask; aliquots of 10 mL, 80% K₂Cr₂O₇ and 15 mL conc. H₂SO₄ were added; mixture was boiled 5 min under reflux and allowed to cool; diluted with water to make to volume. A 30-mL



Figure 1. Sampling locations in Mali (Site 1: 6° 47' W, 12° 55' N; Site 2: 5° 08' W, 12° 11' N; Site 3: 5° 35' W, 11° 14' N).

aliquot of the digest was then diluted to 200 mL; treated with 3 to 4 drops 0.4% diphenylamine, and the remaining dichromate is back titrated with 0.2 N FeSO_4 . The difference between added FeSO_4 for samples compared to a blank titration determines the amount of oxidized C. A correction factor of 1.32 was used to account for incomplete oxidation of organic C (Nelson and Sommers, 1996).

Mass loss on ignition analyses

The loss-on-ignition (LOI) procedure was performed largely as described by Nelson and Sommers (1996). In brief, five gram samples of soil were placed in 50-mL Pyrex beakers that had been pre-weighed for their weight to the nearest 0.1 mg after being conditioned at 400°C for 2 h and then cooled. Beakers containing soil samples were then: 1) placed in a muffle furnace and heated to 105°C for 24 h; 2) cooled to room temperature in desiccators over CaCl_2 ; 3) weight of soil determined to nearest 0.1 mg; 4) placed in a muffle furnace and heated to 400°C for 16 h; 5) cooled in desiccators over CaCl_2 ; and 6) weight of ignited soil determined to the nearest 0.1 mg. The mass of soil loss expressed as a percentage of dry soil weight was then calculated by:

$$\text{LOI (\%)} = (\text{Weight}_{105} - \text{Weight}_{400}) / \text{Weight}_{105} \times 100$$

Thermal analyses

Differential scanning calorimeter (DSC) was performed using a TA Instruments DSC 2920 (TA Instruments, New Castle, DE). During thermal analysis samples (10 mg in aluminum capsules) were analyzed under N_2 purge (20 mL min^{-1}) with a heating rate of $10^\circ\text{C min}^{-1}$ from 20 to 600°C. Before analysis, soil samples were oven dried to 105°C.

X-ray diffraction analysis

Potential changes in soil mineralogy due to mineral decomposition resulting from 400°C heat treatment of soil were assessed by X-ray diffraction (XRD), using a Norelco X-ray diffractometer (North

American Philips Company Inc., Andover, MA) with copper $\text{K}\alpha$ radiation. The X-ray diffraction patterns were obtained using finely ground samples of soil as described by Harris and White 2008. Data are reported as 2-theta (twotheta) in units of degree which can be converted to d spacing (in angstroms) using Bragg's equation:

$$d = \lambda / [2.0 * \sin(0.5 * \text{twotheta} * A)]$$

Where $\lambda = 1.541838$ angstroms ($\text{K}\alpha$ weighted mean wavelength) and $A = 0.0174532$, a constant converting degrees to radians (Bragg, 1913).

Infrared spectral measurements

All samples (air-dried and ground) were scanned in the NIR using a NIRSystems model 6500 scanning monochromator (Foss NIRSystems, Silver Spring, MD) equipped with a rotating sample cup module. Samples were scanned from 400 to 2498 nm with 64 co-added scans taken per sample. Spectra were computed as $\log(1/\text{Reflectance})$ using a ceramic reference for the background spectra. With standard operation of the instrument, spectra were collected from 400 to 1098 nm using a Si detector and from 1100 to 2498 nm using a PbS detector with data collected every 2 nm for a total of 1050 data points per spectra at a nominal resolution of 10 nm.

The samples were scanned in the MidIR from 4000 to 400 cm^{-1} (2500 to 25,000 nm) at 4 cm^{-1} resolution with 64 co-added scans per spectra, on a DigiLab FTS-60 Fourier transform spectrometer equipped with a custom made sample transport which allowed a 50 by 2 mm area sample to be scanned (Reeves, 1996). Absorbance spectra were collected as $\log(1/\text{Reflectance})$ using KBr for the background reference. The "different spectra" in MIR spectral region were calculated by subtracting spectra for ignited soil samples from the corresponding spectra of untreated soil.

Chemometrics

All calibration models were developed using partial least squares regression (PLS) using Galactics PLSPlus running under Figure 2,

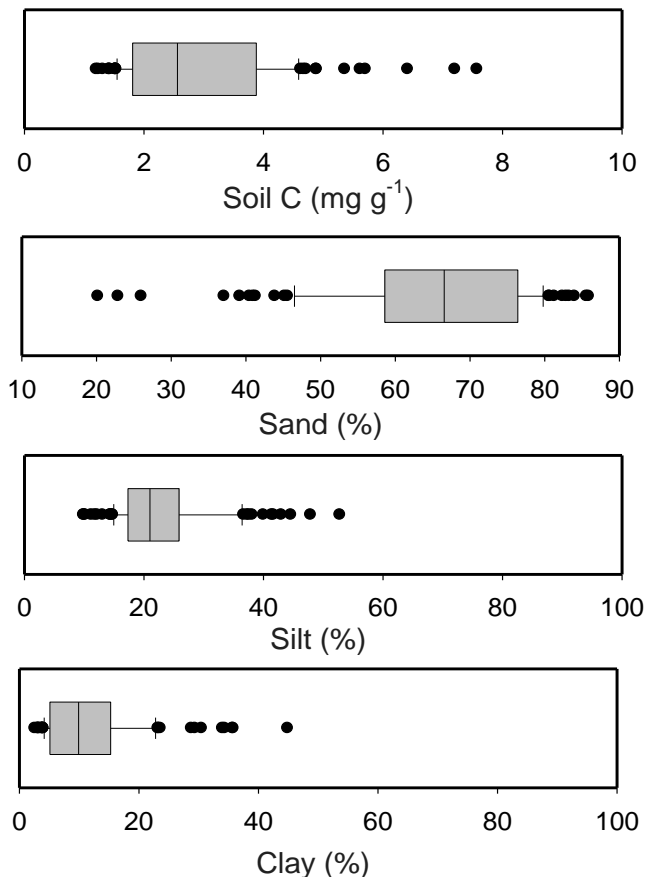


Figure 2. Range and distribution in organic carbon, sand, silt, clay content for soils collected in the Sahel region. The line represents the median, the box represents the 25th and 75th percentile, the whiskers represent 10th and 90th percentile, and the dots represent outlier soil samples.

GRAMS/32 (Galactic Industries, Salem, NH). The number of PLS factors to be used were determined using the F-test from the predicted residual sums of squares error from a one-out cross validation analysis (Massart et al., 1998).

RESULTS AND DISCUSSION

In this study the range of organic C in the West African soils ranged from 1.2 - 7.5 mg g⁻¹ soil with a median value of 2.5 mg g⁻¹ (Figure 2). These low values provide substantial challenge to obtain accurate measurements of soil organic C. The median sand, silt and clay contents were 66, 21 and 10% (g/100 g) respectively. The median clay content in surface soils was 5.1% which compared to 15.2% for subsurface soils. These high sand and low clay contents likely contribute to low soil C contents. One aspect of the study assessed whether ridge tillage management stimulates C sequestration in the Sub-Saharan Africa (Doumbia et al., 2008).

During this study dichromate oxidation was the within-country capability for measurement of soil C. It is well

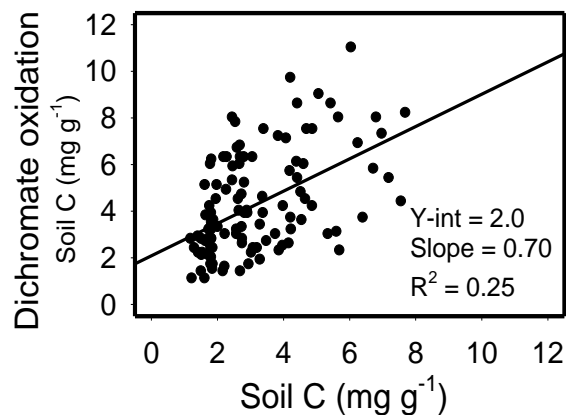


Figure 3. Comparison of dichromate oxidation estimates that were performed within country and dry combustion measurements of soil carbon.

established that this indirect method for estimating organic C is subject to influence by interferences that increase with decreased organic C content and De Vos et al. (2007) determined that the limit of quantification was approximately 4 mg g⁻¹ soil which is well above the median C content within the Sub-Saharan region. The extremely low C content of soils in the Sahel region of West Africa thus provides a substantial challenge to many analytical methods. A comparison of the within-country dichromate method to direct elemental analysis shows substantial deviation from a linear relationship (Figure 3). These findings are in agreement with reported findings that compared analytical results generated with the dichromate method and with dry combustion (Skjemstad et al., 2000; Lettens et al., 2007; De Vos et al., 2007).

Performance issues related to use of dichromate oxidation are reviewed extensively by Nelson and Sommers (1995). In brief, they concluded that procedure is semi quantitative for estimating soil C with reported correction factor ranging from 1.0 - 2.86. This large range in correlation factors may be reflective of the wide range in analytical procedures reported.

Deviations in results may also be caused by well established interferences to dichromate methods. For example, dichromate methods are subject to interferences by certain soil constituents (Nelson and Sommers, 1995). The low C content of soils in this study likely exacerbates the influences of interferences that may be present in the soils.

Our initial assessment of mass loss on ignition as an alternative low-infrastructure method for estimating soil C showed substantial deviation from a linear relationship with elemental analysis when all samples were included in the regression analysis (Figure 4A). However, with segregation by sampling depth, it becomes evident that a tight linear relationship holds for the surface soils (Figure 4B) and the deviations in linear fit occur with the higher

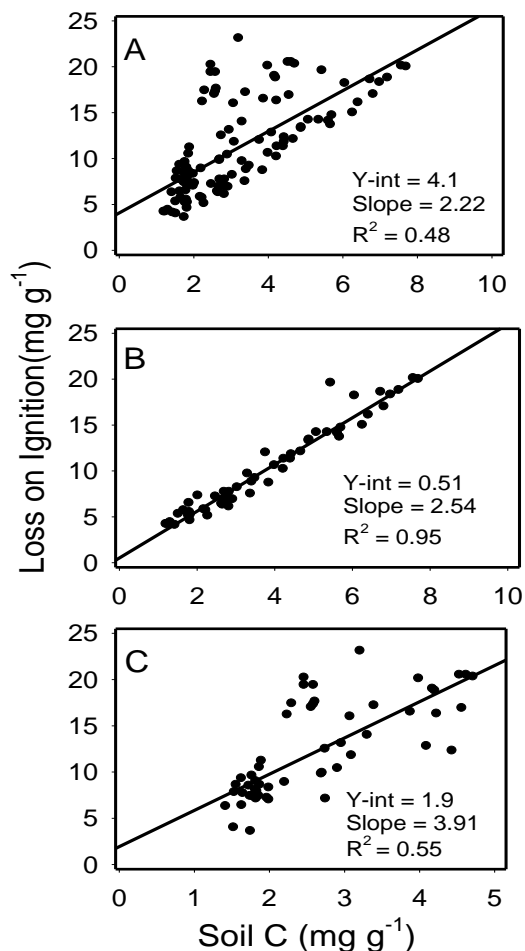


Figure 4. Comparison of loss on ignition to elemental C analysis by dry combustion.

clay subsurface samples and perhaps those that contain more amorphous, water-containing oxides (Figure 4C). Moreover, the slope of the regression lines were substantially different for surface and subsurface samples indicating that these two groups of samples react quite differently to ignition at 400°C.

To investigate differences with sampling depth we performed differential scanning calorimetry (DSC) on paired surface and subsurface samples collected at 5 different locations. Figure 5 shows a typical thermal scan for the paired soil showing that the subsurface soil had a broad endothermic peak at temperatures below 200°C and a sharp endothermic peak at about 300°C. The broad peak below 200°C may be a dehydration involving various forms of bound water. The peak at 300°C is consistent with gibbsite or goethite dehydroxylation (Kloprogge et al., 2002; Prasad et al., 2006) which is a well established interference with loss-on-ignition methodology (Nelson and Sommers, 1995).

XRD analysis (Figure 6) demonstrates the dominance of quartz mineralogy in these sandy soils with the strong

primary peak (d1 spacing) at 26.9° 2 θ (3.3 Å) and probable secondary peaks at 21.2° 2 θ (4.2 Å), 36.7° 2 θ (2.44 Å), and 39.6° 2 θ (2.27 Å). A significant presence of kaolinite is shown by the peak at 12.33° 2 θ (7.17 Å). The 21.2° 2 θ signal could be also be consistent with goethite with reported d1 spacing of 4.18 Å, however there was no evidence of decreases in peak area upon ignition as would be expected with significant goethite decomposition. Additionally, the absence of a significant signal at 18.3° 2 θ associated with the d1 spacing for gibbsite (4.85 Å) indicated little presence of this mineral in these soils. In general, these data show no major shifts in soil mineralogy caused by 400°C ignition indicating that no single type of mineral dehydroxylation was primarily responsible for differences in loss on ignition observed between surface and subsurface soils. Support for a hypothesized more general dehydration is provided by the MidIR spectra of soil samples before (Figure 7A) and after ignition (Figure 7B). The resultant spectra calculated by difference between untreated and ignited samples have features similarity to the spectra of water (Figure 7C) indicating the loss of water upon ignition. It seems likely that the greater surface area in high clay content subsurface soils are more subject to dehydration/dehydroxylation reactions which interfere with loss on ignition analyses. With these limitations, our findings support a conclusion that loss on ignition approaches can perform well for analysis of surface soils in the Sahel provided that they are properly calibrated using elemental analysis. Additionally, the use of different temperatures for soil pretreatment and ignition, than used in this study, may reduce the observed interference associated with dehydration/dehydroxylation reactions.

The utilization of diffused reflectance spectroscopy for measurement of soil C is well established (Janik et al., 1998; Reeves et al., 2001) however the low soil C environment of the Sahel region offers a special challenge. The findings of this study demonstrated an ability to generate strong calibration models for soil C in both the NIR and MidIR spectral regions. Additionally, calibrations for sand, silt and clay were possible (data not shown). These calibrations were developed from soils collected from three geographically diverse fields which give reason to expect that regional calibrations can be developed for soils in the Sahel. The advantages of diffused reflectance methods are that they are rapid and easy to use and can provide multiple analyses with a single scan. In this example accurate measures of soil texture and soil C can be obtained simultaneously. Other advantages are no reagent or waste disposal issues and generally low infrastructure requirements other than the instrument. Because diffused reflectance spectroscopy is a secondary (or indirect) method, the need to build and maintain valid local and regional calibrations should be an ongoing process with the soil calibration set properties always being quantified by the best available primary method of analysis such as dry combustion in the case of

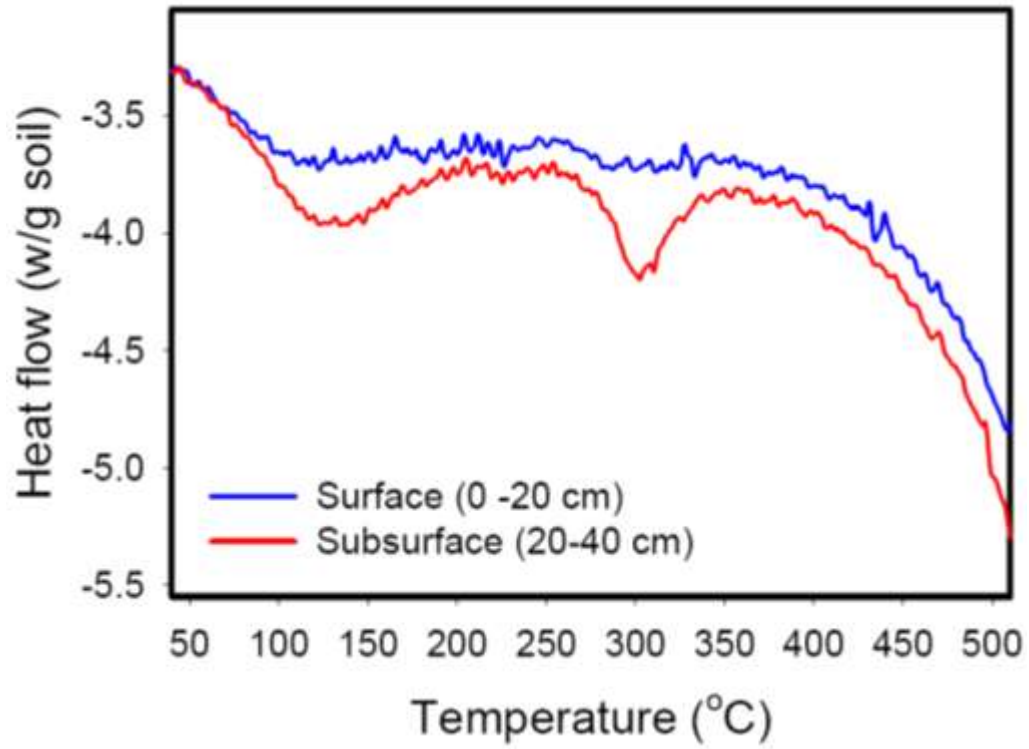


Figure 5. Differential scanning calorimetry (DSC) analysis of surface and subsurface soil samples.

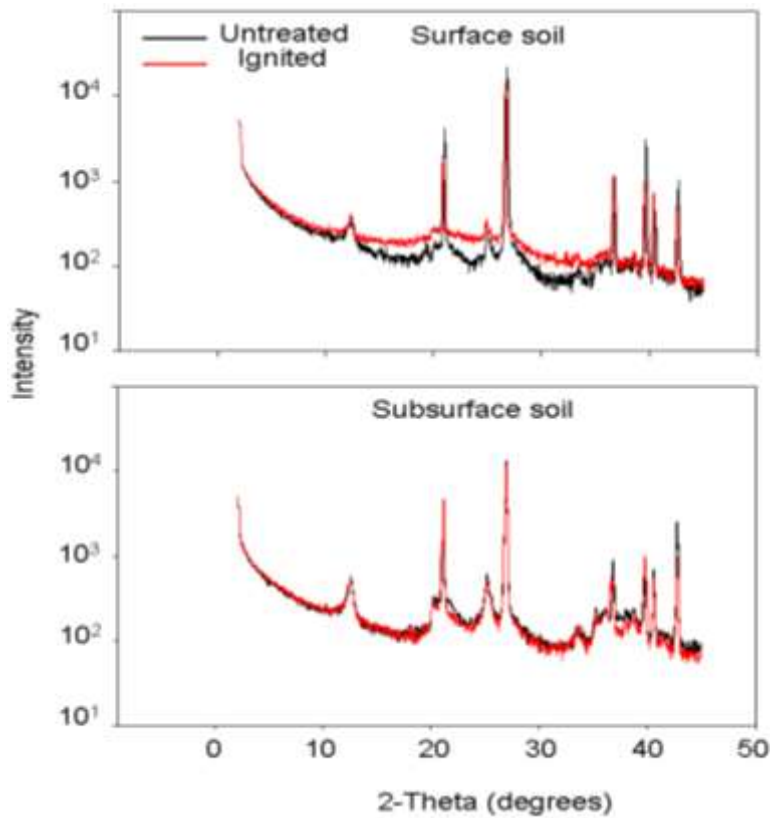


Figure 6. X-ray diffraction (XRD) analysis of surface and subsurface soil samples.

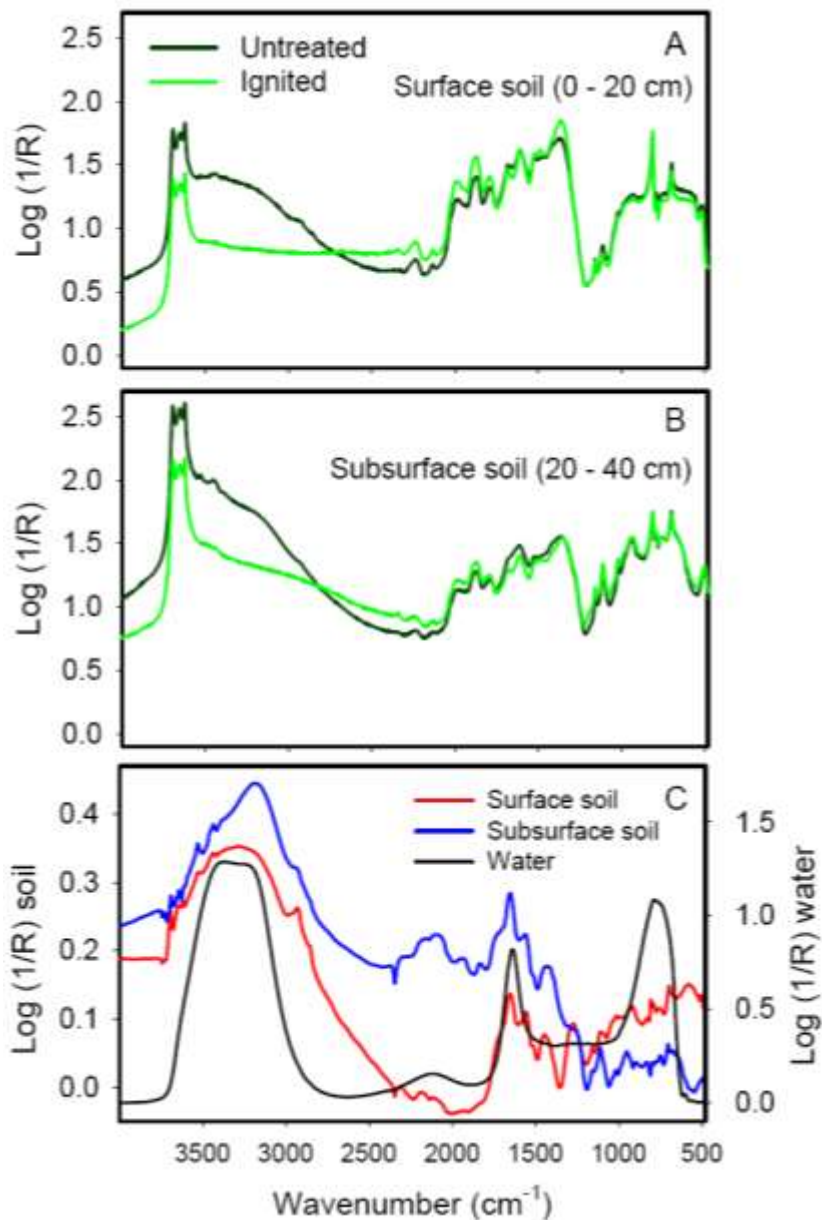


Figure 7. Mid-infrared spectral of (A) surface and (B) subsurface soil before and after ignition at 400°C. The bottom plot (C) shows the difference spectra (untreated – ignited) for both surface and subsurface samples. This plot also contains the spectrum for water for reference.

soil organic C.

Conclusions

In the developing world, most soil analytical laboratories rely on secondary methods of analysis which only provide indirect measurements of soil C and until recently this has also generally been upheld for laboratories in developed countries. With use of such methods, there is considerable risk for analytical drift as methods evolution

as well as general inaccuracy which can provide poor baseline soil C datasets and uncertainty in C sequestration. Our evaluation found that both dichromate oxidation and mass loss on ignition methods can lead to substantial error for soils in Mali which generally have low organic matter content whereas spectroscopic methods hold considerable promise if calibrations are well maintained. With routine use of any indirect method, constant attention is required to check for drift and accuracy by referencing against a primary method of analysis.

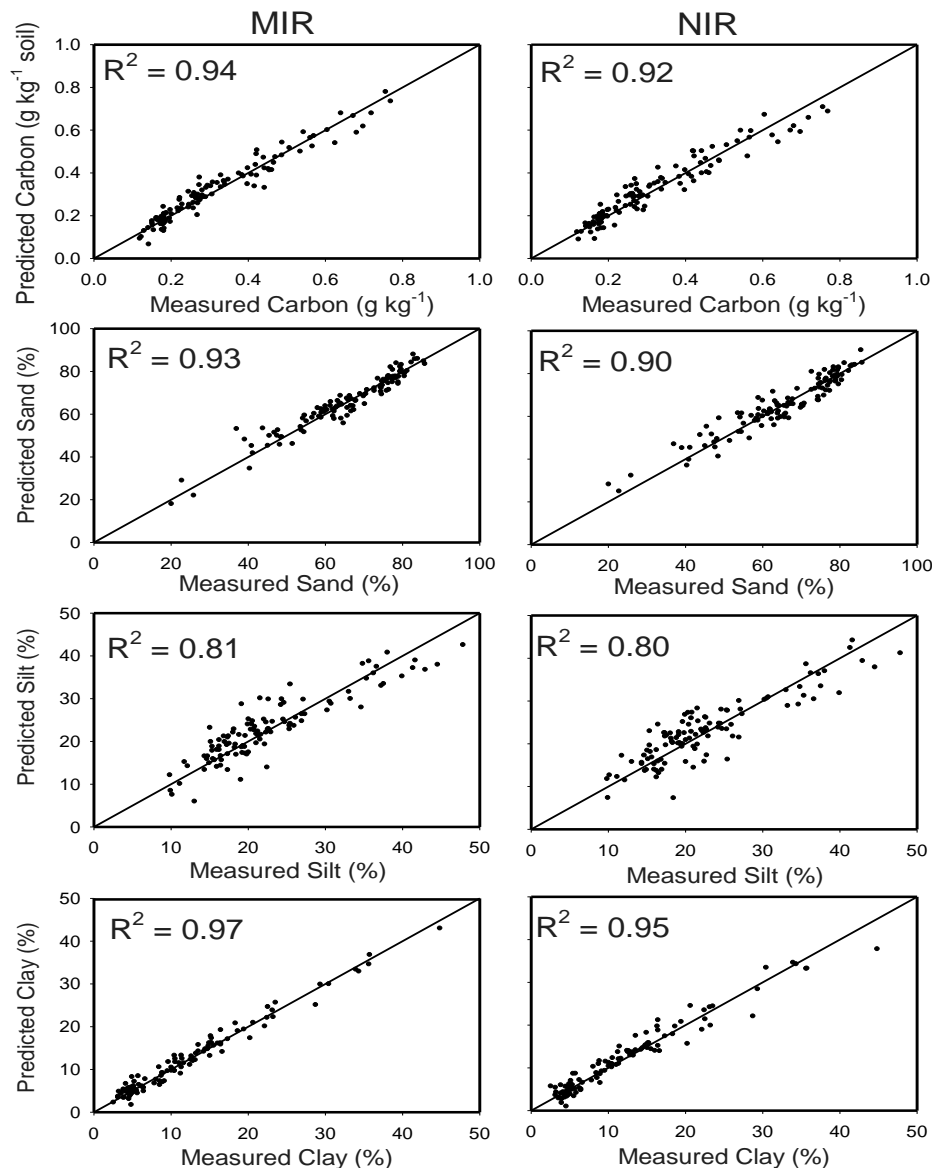


Figure 8. Calibration results for mid infrared (MIR) and near infrared (NIR) diffuse reflectance spectroscopy for soil C, sand, silt and clay.

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