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► **To cite this version:**

Bernard G. Barthès, Jean-Luc Chotte. Infrared spectroscopy approaches support soil organic carbon estimations to evaluate land degradation. *Land Degradation and Development*, 2021, 32 (1), pp.310-322. 10.1002/ldr.3718 . hal-02936620

HAL Id: hal-02936620

<https://hal.inrae.fr/hal-02936620>

Submitted on 30 May 2023

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Infrared spectroscopy approaches support soil organic carbon estimations to evaluate land degradation

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Abstract

Soil organic carbon (SOC) is an acknowledged indicator for land degradation, but conventional determination of SOC remains tedious, especially regarding SOC stock (in kgC m^{-2} for a given depth layer), which is the product of SOC concentration (gC kg^{-1}) by volumetric mass (kg dm^{-3}). Diffuse reflectance infrared spectroscopy (DRIS) is a time- and cost-effective approach, which uses calibrations for making predictions. The aim of this paper is to propose an overview of DRIS uses for estimating SOC, thus land degradation. Indeed, many papers have demonstrated the precision of DRIS for quantifying SOC concentration, at different scales. Current development of large soil calibration databases and improvements in spectral data analysis pave the way for ever-wider use of DRIS, which should help solving the soil data crisis, regarding SOC especially. The increasing availability of portable spectrometers allows SOC quantification in the field, which seems particularly promising; but large calibration databases made of soil spectra acquired in the field are difficult to build, while large collections of analyzed soil samples (air dried, 2-mm sieved) already exist. Some recent studies indicate that DRIS can also be used for predicting SOC stock, even from sieved samples, which represents an efficient option because determining the volumetric mass is particularly tedious and an obstacle for exactly specifying the role of soils in the global carbon cycle. In short, DRIS has strong potential for supporting better evaluation of soil and land degradation, and the availability of spectrometers at increasingly affordable prices reinforces this potential.

Keywords

Near infrared reflectance spectroscopy (NIRS) – Mid-infrared reflectance spectroscopy (MIRS) – carbon concentration – carbon stock – multivariate regression

1. Introduction

Land degradation represents huge challenge. It consists in the reduction of biological or economic productivity and complexity of agroecosystems as a result from combined pressures, including land use and management practices (UNCDD, 1994). This phenomenon is considered pervasive and systemic, with negative impacts on biodiversity and ecosystem services (IPBES, 2018) and on climate change (IPCC, 2019). The scientific community has provided evidences on the drivers of land degradation (Mbow et al., 2015; Tully et al., 2015; Sklenicka, 2016; Safriel, 2017) and on means to combat it (de Vente et al., 2016; Kust et al., 2017; Sanz et al., 2017; Felix et al., 2018; Giger et al., 2018). In September 2018, the General Assembly of the United Nations adopted the Sustainable Development Goals, which, in particular, aim at restoring degraded lands and soils and at achieving a land degradation neutral world by 2030 (Target 15.3; United Nations, 2018). This represents a clear political recognition of the key role of soils and lands to achieve sustainable development. Land Degradation Neutrality (LDN) has been defined as "a state whereby the amount and quality of land resources necessary to support ecosystem functions and services and enhance food security remains stable or increases within specified temporal and spatial scales and ecosystems" (UNCCD, 2016). A scientific conceptual framework has been developed to implement LDN and provide guidance to monitor progress to LDN (Cowie et al., 2018). For these purposes, degradation is assessed through three indicators as proxies for the capacity of land to deliver ecosystem services: land cover, land productivity, and soil organic carbon (SOC).

Indeed, SOC is the main constituent of soil organic matter, which has a long-acknowledged and key role in soil fertility through nutrient release, structure stabilization, improvement in water-holding capacity and soil biodiversity, biodegradation of contaminants, etc. (Lal, 2006). Moreover, SOC is the largest terrestrial carbon pool, and is thus an important component in the global carbon cycle (Lal, 2019). Organic carbon is present in the soil in a wide range of chemical forms: carbohydrates, proteins and protein-derived compounds, lipids, phenols and cyclic nitrogenous compounds, and still unknown compounds (Paul, 2016). Analyzing the exact molecular composition of organic matter in soils is hardly feasible due to its complex nature. So SOC has often been separated into fractions having homogenous characteristics (Chenu et al., 2015): firstly alkali/acid solubility pools (fulvic acids, humic acids and humin); then more functional pools, for instance kinetic pools, having specific turnover rates (labile, stable, resistant), or chemical functional groups (alkyl, O-alkyl, aromatic, carbonyl, etc., determined by nuclear magnetic resonance). Studying such SOC fractions allows deep

understanding of SOC composition, dynamics and stability as affected by climate, mineralogy or land use and management. However, studying such SOC fractions is not necessarily the most appropriate level for assessing land degradation, especially when looking at practical indicators. Actually, total SOC is considered by both scientists and farmers a primary indicator of soil quality and health, which itself is considered a primary indicator of sustainable land management (Doran, 2002). When dealing with land degradation practically, it therefore seems relevant to focus on total SOC (simply denoted SOC hereinafter).

Conventional quantification of SOC requires analyzing SOC concentration, which is generally carried out by Dumas combustion or sulfochromic oxidation, using reagents and producing wastes that are toxic (Pansu & Gautheyrou, 2006). Moreover, SOC concentration (in gC kg^{-1}) is not an additive variable, thus quantifying SOC at field, region or country scale involves determining the apparent volumetric mass (or bulk density, D_b , in kg dm^{-3}), in order to calculate SOC stock (in gC dm^{-3} , or in kgC m^{-2} for a given soil layer), which is additive (Poeplau et al., 2017). The determination of D_b is generally achieved by drying at 105°C then weighing a known volume of intact soil (with undisturbed structure), which is most often collected using a bevelled cylinder thus requires digging a pit or using a mechanized coring device. Quantifying SOC is thus tedious using conventional approaches, especially for SOC stock.

In recent decades, increasing attention has been paid to diffuse reflectance infrared spectroscopy (DRIS) for characterizing soil properties, SOC concentration especially (Dalal & Henry, 1986; Sudduth & Hummel, 1991; McCarty et al., 2002; Barthès et al., 2006; Bellon-Maurel & McBratney, 2011). Indeed, DRIS is a time- and cost-effective, non-destructive and reproducible approach, with no reagent required and no waste generated; moreover a single spectrum may be used to assess various properties. In its current uses DRIS is based on calibration, using multivariate regression and developed on calibration samples characterized by both spectral data and conventional reference data; then calibration can be applied to predict the property considered on new samples only from their spectrum. Indeed, the property is not measured but predicted, with known uncertainty, quantified using standard error of prediction. As stated by Reeves (2010), conventional analytical approaches produce small amounts of highly accurate, expensive data, while DRIS produces large amounts of low-cost, fair quality data, with specified uncertainty. In this way, McBratney et al. (2006) considered the combination of DRIS and multivariate statistics an opportunity for solving the soil data crisis.

This paper proposes an overview to demonstrate the relevance of DRIS for high-speed and low-cost quantification of SOC concentration and stock. So, given the key role of SOC in soil fertility and in the global carbon cycle, we aim at demonstrating that DRIS represents great opportunity for evaluating the land capacity to deliver ecosystem services.

2. DRIS quantification of SOC concentration

2.1. Physical background and historical outlines

Infrared radiation causes molecular bonds to vibrate, and the vibrations absorb energy at wavelengths that depend on bond strength, on the atoms involved, and on chemical environment (Siesler, 2007). So when compared with the incident radiation, the radiation reflected by a solid sample is modified in the spectral regions that correspond to energy absorption by the sample. Fundamental vibrations occur in the mid infrared region ($4000\text{--}400\text{ cm}^{-1}$, i.e. $2500\text{--}25,000\text{ nm}$), while their overtones and combinations mostly vibrate in the near infrared region ($800\text{--}2500\text{ nm}$), where they overlap (Bellon-Maurel & McBratney, 2011). Reflected spectra thus display much clearer peaks in the mid than in the near infrared range (Stenberg et al., 2010; cf. Figure 1), which explains why the former have long been used for molecule identification, based on visual observation. For instance, the regions around $2750\text{--}2670$ and $1730\text{--}1710\text{ cm}^{-1}$ have been assigned to saturated aliphatic carboxylic acids, and the region around 1650 cm^{-1} to amides (Socrates, 2001; Barthès et al., 2016). Such peak recognition is hardly possible with near infrared spectra (except for water, cf. Figure 1), though spectral regions could also be assigned to chemical compounds (Workman & Weyer, 2008; Barthès et al., 2016). In general statistical tools are required for analyzing near infrared spectra, hence the more recent applications of this spectral range for analytical purposes. However, absorption is much stronger in the mid than in the near infrared range (cf. Figure 1), according to the vibrations they are associated with (i.e. fundamentals vs. overtones, respectively; Bellon-Maurel & McBratney, 2011). As a consequence, the reflected radiation is stronger in the near infrared, thus requires less sensitive and less costly sensors. This explains the rapid development of quantitative applications of near infrared spectroscopy once computer and data analysis sciences developed, followed, after a while, by their counterparts in the mid infrared range.

Numerous authors have reported the usefulness of DRIS for characterizing SOC concentration based on spectra acquired in laboratory conditions (most often on air dried, 2-mm sieved samples). Bowers & Hanks (1965) were the first to demonstrate the qualitative effect of SOC on visible and near infrared reflectance (VNIR) spectra by comparing the same soil sample

with and without SOC. Quantification of SOC concentration from NIR spectra was firstly achieved by multiple linear regression, using a few wavelengths, then by principal component regression (PCR) and partial least squares regression (PLSR), which are more sophisticated procedures, based on latent variables (i.e. small numbers of orthogonal variables inferred from large numbers of redundant observed variables, such as infrared spectra, and used for replacing them in regression procedures; Dalal & Henry, 1986; Sudduth & Hummel, 1991). Similar PLSR approach was then used for inferring SOC concentration from mid infrared reflectance (MIR) spectra (Janik et al., 1995) Since then, numerous papers have used PLSR to infer SOC concentration from VNIR, NIR or MIR spectra; while some authors used nonlinear regression procedures such as local regressions (predictions made only using spectral neighbours) or data mining approaches like neural networks, boosted regression trees, random forests, etc. (Stenberg et al., 2010; Ramirez-Lopez et al., 2013; Vågen et al., 2016); and others tested spiking, which consists of enriching the calibration database with samples from the target set (Guerrero et al., 2010, 2014).

Assessing the fit between predictions and observations has often been achieved using R^2 , which is widely known and, to some extent, allows comparison between different studies. However, R^2 represents proportionality rather than similarity, thus may sometimes result in inappropriate judgement (though this has rarely been the case in the domain considered). The root mean square error (RMSE, often called standard error of prediction) is more specific but not very informative as long as the variable distribution has not been presented; moreover it hardly allows convenient comparisons between studies. The RPD (ratio of performance to deviation) is the ratio of observed standard deviation to RMSE, which integrates information on the variable distribution thus can be used for comparing the predictions of different variables, or different studies. Bellon-Maurel et al. (2010) however considered RPD inappropriate for variables that do not have normal distribution, and instead, recommended RPIQ (ratio of performance to interquartile distance, which is the ratio of interquartile distance to RMSE). Nevertheless, RPIQ has not been widely used to date, so RPD has remained useful for comparison between studies, and we have used it hereinafter, along with R^2 . According to Chang et al. (2001), predictions of soil properties were considered accurate when $RPD \geq 2$, and acceptable when $1.6 \leq RPD < 2.0$.

2.2. Large calibration databases

In their review on SOC quantification by DRIS, Bellon-Maurel & McBratney (2011) insisted on the need for as large and exhaustive calibration databases as possible. Some work had

already be done in the VNIR range by Shepherd & Walsh (2002) for eastern and southern Africa, by Brown et al. (2006) for the USA, and other continents to a lesser extent, and by Viscarra Rossel & Webster (2012) for Australia (Table 1). These studies gathered samples from different experiments, trials and surveys. More systematic spectral databases have then been presented, which aimed at covering areas relevant from geographical or administrative viewpoints. Noticeable work has been carried out in France using the national soil quality monitoring network and its associated bank of soil samples collected regularly over the French metropolitan territory (Arrouays et al., 2002), either using the VNIR, NIR or MIR range, and topsoils only or both top- and subsoils (Gogé et al., 2012, 2014; Grinand et al., 2012; Clairotte et al., 2016; Table 1). Several authors have also worked with the LUCAS (Land use/cover area frame statistical survey; Tóth et al., 2013) VNIR database, which represents the topsoils from 23 European Union countries, more or less densely sampled. They distinguished cropland, grassland, woodland, other mineral soils and organic soils, possibly used covariables, and either performed data mining approaches (Stevens et al., 2013) or local PLSR (Nocita et al., 2014). Interestingly, local PLSR yielded better predictions than data mining approaches without covariable, but worse results when using sand as covariable (Table 1); moreover, using geographical coordinates as covariables did not improve the results (Nocita et al., 2014). Other large VNIR calibration databases have been built in China (Shi et al., 2015, who improved predictions when using spectral but also geographical neighbours for calibration) and Australia (Lobsey et al., 2017, who performed local calibration with independent validation in Australia but also New Zealand; Table 1). An attempt has even been made to build a global VNIR library, using samples from 92 countries (Viscarra Rossel et al., 2016a). Though the diversity of conventional methods used for analyzing SOC concentration (dry combustion, wet oxidation, loss on ignition, etc.), predictions using a machine learning algorithm with spectral pre-classification were accurate. Table 1 shows that most studies achieved accurate predictions of SOC concentration using large soil spectral libraries, with R^2_{val} and RPD_{val} around 0.8 and > 2 in general, and up to 0.9 and > 3 sometimes, respectively, even using common regression approach (PLSR). When procedures were optimized, in particular the representativeness of calibration samples (e.g. using spectral neighbours) and the spectral range (e.g. MIR for soils from temperate regions, cf. 2.5), standard error of prediction close to standard error of laboratory reference analysis could be achieved (e.g. 2.0 vs. 1.0-1.5 g kg⁻¹ according to Clairotte et al., 2016, and Stevens et al., 2013, respectively). In such conditions, DRIS could helpfully be used in SOC estimation and monitoring for LDN planning; but to our knowledge, this has not been the case yet.

2.3. Types of soil samples used for spectrum acquisition

Spectrum acquisition in laboratory conditions is generally made on air-dried, 2-mm sieved soil sample, which is the most general soil preparation for laboratory analyses and for long-term sample conservation (Pansu et al., 2001). However, the sample may be further prepared for specific analyses or purposes; for instance SOC analysis by dry combustion is made on finely ground sample (e.g. < 0.2 mm; Pansu & Gautheyrou, 2006). Studies that compared NIRS predictions made on coarsely vs. finely prepared samples indicated in general that fine grinding (from < 0.2 to < 0.5 mm) resulted in better prediction of SOC concentration than coarse preparation (from < 2 to < 4 mm) in clayey soils but not in coarse-textured soils (Dalal & Henry, 1986; Fystro, 2002; Reeves et al., 2002; Russel, 2003; Barthès et al., 2006; Brunet et al., 2007). These contradictory effects have been explained by the size and arrangement of particles, which affect light transmission (Chang et al., 2001; Barthès et al., 2006). More accurate MIRS prediction of SOC concentration using spectra acquired on 0.2- than on 2-mm samples is a less controversial result, though it has rarely been studied (Barthès et al., 2016). Actually, to our knowledge, all studies carried out to date in laboratory conditions (except the above-mentioned) have used finely ground samples for MIR spectroscopy (< 0.2 mm and sometimes finer), which indicates there is implicit agreement that MIR requires finely ground samples.

Moreover, several authors reported more accurate VNIRS prediction of SOC concentration on air-dried cores (a few dm in size) or clods (a few cm in size) than on air-dried, 2-mm sieved samples, possibly because higher bulk density in clods or cores would result in higher reflectance (Morgan et al., 2009; Fontán et al., 2010).

Gras et al. (2014) even achieved more accurate VNIRS prediction of SOC concentration on field-moist than on air-dried clods, probably due to better light transmission. Indeed, increasing moisture reduces the refraction difference between soil particles and pore spaces, thus increases forward light scattering, hence the probability of photon absorption by soil particles (Twomey et al., 1986). Actually, several authors reported similar or better VNIRS performance in field than in laboratory conditions (cf. Table 2), which is counterintuitive a priori because, among other things, field acquisition involves variable soil moisture and the presence of coarse particles (which are removed before reference analyses used for calibration). But field conditions might be beneficial to some respects due to higher sample density, and higher cohesion in moist conditions (Stevens et al., 2008; Gras et al., 2014). However, Morgan et al. (2009) achieved less accurate VNIRS prediction of SOC

concentration on field-moist cores than on air-dried, 2-mm sieved samples, probably because field-moist cores had contrasting moisture contents (sampling had been performed in rather hot conditions with possible storms or irrigation, and at different depths). Veum et al. (2018), Allory et al. (2019) and Hutengs et al. (2019) similarly reported less accurate SOC prediction in field than in laboratory conditions firstly due to variable in-situ soil moisture content: moisture conditions might vary among depth layers (when several were sampled) and/or during long field campaigns (Table 2). In the study reported by Morgan et al. (2009), prediction accuracy ranked as follows: air-dried cores > air-dried 2-mm sieved > field-moist cores. While for Gras et al. (2014), also on a range of soils, the rank was: field-moist cores > field-moist clods > air-dried clods > air-dried 2-mm sieved. In that study, field-moist samples had rather homogeneous soil moisture because all had been collected in wet conditions in winter. In laboratory conditions, Stenberg (2010) also observed that VNIRS predictions of SOC concentration were more accurate on standardized rewetted than on air-dried samples, all sieved at 2 mm.

These results suggest the following trends:

- prediction accuracy increases with sample particle size, from finely ground samples to cores, due to better light transmission; except on ground clayey soils, for which prediction is better on 0.2- than on 2-mm samples, probably due to higher homogeneity and lower diffusivity of finely ground clayey samples;
- prediction is better on moist than on dried samples when moisture is homogeneous among samples, due to better light transmission, but this is the opposite when moisture content varies among samples, because varying moisture brings noise to the spectra; thus field spectroscopy would be recommended outside dry periods and rainfall (or irrigation) events;
- prediction is better on 2-mm sieved than on coarsely prepared soil samples (clods, etc.) when the soil is rich in coarse particles (> 2 mm), and the difference increases with their proportion.

These trends have to be confirmed with other studies that would compare different sample preparations, moistures and coarse particle contents, on large and diverse sample sets as far as possible.

2.4. Spectrum acquisition in laboratory vs. field conditions

As seen above, spectrum acquisition on field moist samples, possibly cores or clods (coarsely crushed cores) may yield accurate predictions. Getting soil spectra directly onsite, in the field, has aroused much interest because this eliminates the need for sample packaging, transport,

drying, crushing and sieving, except for calibration purposes. This allows characterizing many samples and improves even more time- and cost-effectiveness (Viscarra Rossel et al., 2011). However, prediction of soil attributes using spectra acquired in the field most generally requires, to date, calibration with spectrum acquisition in the field, which is more tedious than spectrum acquisition in the lab. This explains why much less applications to soils have been carried out in the field than in the laboratory. Indeed, collections of conventionally analyzed soil samples are available air-dried and sieved (i.e. ready to be scanned), but no such collection of analyzed samples is available with samples as they were in the field. So calibration of field spectra requires specific field campaign and conventional analyses, which is particularly tedious and costly if the calibration database has to be representative of a country.

Nevertheless some work has been done. Sudduth & Hummel (1993) were the first to report NIR spectrum acquisition in the field. Then a range of studies reported good predictions of SOC concentration at either local, regional or multi-regional scale, using VNIR spectra acquired in boreholes, on fresh cores, on crushed fresh samples, from the soil surface, from a measurement height of 1 m, or on-line with a spectrometer probe fitted to a subsoiler (Mouazen et al., 2006; Stevens et al., 2008; Kusumo et al., 2008, 2011; Nocita et al., 2011; Gras et al., 2014; Cambou et al., 2016; Nawar & Mouazen, 2018, 2019; Allory et al., 2019; Hutengs et al., 2019; Pei et al., 2019). Prediction results were often good, but not as good in general as in laboratory conditions ($R^2 = 0.71$ to 0.87 and $RPD = 1.9$ to 2.8 in cross- or external validation; Table 2). However, less accurate in situ VNIR predictions of SOC concentration have sometimes been reported (Morgan et al., 2009; Veum et al., 2018; Nawar & Mouazen, 2019; $R^2_{val} = 0.64-0.65$ and $RPD_{val} = 1.5-1.7$), and even poor ones (Hutengs et al., 2019; $R^2_{val} = 0.27$ and $RPD_{val} = 1.2$). This might be due to variable moisture content among samples (cf. 2.3) or insufficient representativeness of calibration samples (e.g. selected at random).

Much less field work has been carried out using MIRS, because transportable MIRS is recent technology. In an exploratory study, Izzaualde et al. (2013) achieved good SOC predictions from MIR spectra of samples collected at different depths in a field, using calibration samples from that field, from another field and from archived samples ($R^2_{val} = 0.77$ and $RPD_{val} = 2.1$). Ji et al. (2016) studied two fields using MIR spectra acquired at 5-cm depth, and achieved good SOC predictions for one field, where the soil was very organic, but poorer ones for the other field, less organic ($R^2 = 0.86$ and 0.60 and $RPD = 2.6$ and 1.6 in cross-validation, respectively). Hutengs et al. (2019) studied 90 fields (one sample per field) in a small region

and achieved acceptable SOC predictions ($R^2_{\text{val}} = 0.63$ and $\text{RPD}_{\text{val}} = 1.7$). According to the authors, variable soil moisture content during field campaign (in spring) affected prediction accuracy negatively; but the fact that MIR spectra had been acquired from the soil surface while SOC was analyzed at 0-5 cm depth might also be considered (Table 2).

Developing tools for applying calibrations built in laboratory conditions (on air-dried, 2-mm sieved samples) to spectra acquired in the field (moist, structured, with coarse particles, etc.) represents a great challenge and would have important consequences. Indeed, this would allow applying easy-to-build calibration databases made from archived soil collections to easy-to-get field spectra. Some authors have begun trying to remove the effects of moisture and structure from spectra, by identifying the spectral subspaces they affect then projecting the spectra orthogonally. Minasny et al. (2011) studied 2-mm sieved samples at different moisture contents and reported that VNIRS prediction accuracy of SOC concentration, instead of being deteriorated for moist samples (R^2_{val} decreased from 0.83 for air-dried samples to 0.56 for wet samples, and RPD_{val} decreased by 70%), was similar whatever sample moisture content after spectrum "orthogonalization". Ge et al. (2014) studied intact cores and 2-mm sieved samples either air-dried or wet, and observed that VNIRS prediction of SOC concentration using wet cores was improved after orthogonalization (R^2_{val} increased from 0.49 to 0.53 and RPD_{val} increased by 30%). This was mainly due to the removing of moisture effect, while the effect of structure was more difficult to address. Such approaches require scanning samples in different moisture and structural conditions, which is tedious, and has been limited to rather small sample sets to date.

2.5. Spectral range: (V)NIRS vs. MIRS

Many papers have reported better prediction of SOC concentration using MIR than (V)NIR spectra (Reeves et al., 2001, 2002; McCarty et al., 2002; McCarty & Reeves, 2006; Viscarra Rossel et al., 2006; Igne et al., 2010; Dong et al., 2011; Xie et al., 2011); but it is worth noting that they concerned temperate regions (except Dong et al., 2011), and rather homogeneous soil sample sets in general. The trend was less clear in other studies (Madari et al., 2005; Yang et al., 2012), while some even reported better NIRS than MIRS predictions (Madari et al., 2006; Ludwig et al., 2008; Rabenarivo et al. 2013; Barthès et al., 2016; and also Shao & He, 2011, for N concentration), regarding tropical and subtropical regions (except Ludwig et al., 2008) and more diverse sample sets.

The supposedly superior performance of MIRS has been attributed to the richer information it provides (cf. Figure 1): more defined peaks, assigned to fundamental vibrations; while NIR is

the range of overlapping overtones and combinations (Reeves, 2010; Bellon-Maurel & McBratney, 2011). However, in Al and Fe oxide-rich soils (i.e. in particular tropical and subtropical regions), MIR information relating to SOC could be partly masked due to the overlap of regions relating to mineral and organic components (e.g. metal oxides often absorb at 1020-970 and 1100-825 cm^{-1} while carbohydrates and polysaccharides absorb at 1080-1030 and 1170-950 cm^{-1} , respectively; Rabenarivo et al., 2013). And this would explain better NIRS than MIRS prediction of SOC in tropical and subtropical regions. But this result has to be consolidated with other studies, on large and diverse sample sets as far as possible.

Importantly, Barthès et al. (2016) reported noticeably poorer MIRS predictions using spectra acquired on 2- than on 0.2-mm samples; thus MIRS requires fine grinding, which is somewhat tedious and contradicts the time- and cost-effectiveness of the approach. In contrast, these authors found little difference in SOC prediction accuracy when NIR spectra were acquired on 2- vs. 0.2-mm samples. This is confirmed by the fact that all cited MIRS applications used finely ground soil (< 0.2 mm or finer), while most cited NIRS applications used more coarsely-prepared soil (< 2 mm in general).

3. DRIS quantification of SOC stock

Sample SOC stock (gC dm^{-3}) is calculated as the product of SOC concentration (gC kg^{-1}) and apparent volumetric mass (or bulk density, D_b , kg dm^{-3}), preferably weighted by the proportion of fine earth (< 2 mm; Poeplau et al., 2017); and then reported for a given depth layer (kgC m^{-2} or MgC ha^{-1} for that depth layer).

Some studies have attempted to estimate SOC stock from measurement of D_b and DRIS prediction of SOC concentration: Cardinael et al. (2015) used VNIR spectra acquired on field-moist intact cores, and Baldock et al. (2018) used MIR spectra acquired on air-dried, 2-mm sieved sample; but these studies presented predictions of SOC concentration, not SOC stock. Moreover, conventional determination of D_b is tedious, as it involves collecting samples of known apparent volume, which requires opening pits or using hydraulic coring machine. Some studies used other approaches to determine D_b . Priori et al. (2016) studied samples from Sicilian fields, and besides VNIRS prediction of SOC concentration on air-dried, 2-mm sieved samples, they predicted D_b using a pedotransfer function that involved measured SOC concentration and texture. These authors achieved $R^2_{\text{val}} = 0.77$ and $\text{RPD}_{\text{val}} = 2.1$ for SOC stock prediction on fine earth (< 2 mm), which was then corrected for semi-quantitatively-estimated volumetric gravel content. On field-moist intact cores, Lobsey & Viscarra Rossel (2016) and Viscarra Rossel et al. (2016b, 2017) used gamma-ray attenuation for predicting

D_b , which was corrected for volumetric moisture content using VNIR spectra; and spectra were also used for predicting SOC concentration. However, these authors aimed at evaluating SOC stock at the field level, and did not present comparisons between observed and predicted SOC stocks at the sample or profile level (except Viscarra Rossel et al., 2016b, who predicted SOC stocks at the profile level using kriging in addition to gamma-ray attenuation and VNIRS, which added a layer of uncertainty: in leave-one-out cross-validation, they achieved $R^2 = 0.4-0.5$ and $RPD = 1.3-1.4$). Attempts to directly predict D_b using DRIS have rarely been successful: several studies obtained poor predictions ($R^2_{val} < 0.3$ and $RPD_{val} < 1.2$; Minasny et al., 2008; Moreira et al., 2009; Veum et al., 2015). Some authors reported good figures of merit ($R^2_{val} = 0.64-0.81$ and $RPD_{val} = 1.7-2.2$; Askari et al., 2015; Roudier et al., 2015; Cambou et al., 2016; Pérez-Fernández & Robertson, 2016). But this might sometimes have been due to homogenous soil population (Roudier et al., 2015), or conversely to very wide distribution (prediction error being actually larger than in other studies; Pérez-Fernández & Robertson, 2016), or to aggregation of sites with poor within-site prediction (Cambou et al., 2016).

Actually, in their review Bellon-Maurel & McBratney (2011) recommended direct DRIS prediction of SOC stock, and considered works in that direction should be a priority. And indeed, doing so, some authors have got promising results. Roudier et al. (2015) achieved good predictions of sample SOC stock in one large arable field, using VNIR spectra from fresh intact cores taken with hydraulic coring equipment mounted on a truck ($R^2_{val} = 0.85$ and $RPD_{val} = 2.6$). Cambou et al. (2016) achieved acceptable prediction of sample SOC stock in two agroforestry fields 250 km apart using VNIR spectra of fresh cores collected using handheld auger ($R^2_{val} = 0.70$ and $RPD_{val} = 1.8$), which is a much lighter approach thus opens exciting prospects. Cores collected with such manual auger are made of partly disturbed soil material; but the results indicate they nevertheless contain information on structure and SOC stock, which can be extracted from their VNIR spectra. Of course, information on SOC stock is poorer than from intact soil materials, which explains why prediction was less accurate in the latter study than in the former (cores with disturbed vs. undisturbed soil material, respectively); along with the probable fact that the homogeneity of the soil sample set was lower in the latter study than in the former (two agroforestry sites in undulating loess areas 250 km apart vs. one arable site in a sand plain, respectively), which often makes DRIS predictions less accurate (Brunet et al., 2007). Allory et al. (2019) also scanned intact soil material but on pit walls, and achieved good VNIR prediction of sample SOC stock in urban soils of two French cities 800 km apart ($R^2_{val} = 0.78$ and $RPD_{val} = 2.2$), though marked

differences between soils (calcareous or acid soils, under parks or streets). Unexpectedly, these authors achieved noticeably more accurate sample SOC stock prediction from spectra acquired in laboratory conditions (air-dried, 2-mm sieved samples; $R^2_{\text{val}} = 0.89$ and $\text{RPD}_{\text{val}} = 3.1$), probably due to variable soil moisture during in situ spectrum acquisition. Accuracy was even improved when SOC stock was considered at the profile level (in kgC m^{-2} ; $R^2_{\text{val}} = 0.94$ and $\text{RPD}_{\text{val}} = 4.4$) than at the sample level (in gC dm^{-3}), probably because some uncertainties at the sample level could offset each other at a more integrated level.

4. Conclusions and perspectives

Many studies have evidenced the usefulness of DRIS for quantifying SOC concentration, especially when using large calibration databases (e.g. national spectral databases), with R^2 up to 0.9 and RPD up to 3.3 in external validation. And it is likely that improvements in spectral data analysis will allow even more accurate predictions, in particular through approaches such as local calibration and spiking. Complementary use of soil expert knowledge could also be fruitful, for instance for calibrations based on soil textural or mineralogical classes, or using covariables easy to obtain (e.g. sand content).

Though more and more portable instruments allow implementation in the field, it may be assumed that most DRIS applications to soils will still long be carried out in laboratory conditions. Indeed, building calibration databases with spectra acquired in the field represents huge work, moreover field spectra may be acquired according to different procedures (on the soil surface, on clods, on cores, on pit walls, etc.) and spectra acquired in different conditions cannot easily be grouped in a common calibration database. In contrast, large calibration databases using soil spectra acquired in laboratory conditions are already available, will very probably become larger, and are often based on one type of soil samples (i.e. air-dried, 2 mm sieved). Developing tools that would allow using calibration databases built in laboratory conditions for making predictions from field spectra is a very exciting perspective; but according to the results achieved to date, such solution will not be operational soon.

As emphasized by Bellon-Maurel & McBratney (2011), direct DRIS prediction of SOC stock represents an efficient option, using spectra acquired in field conditions, or more simply, in laboratory conditions. Indeed, determining SOC stock without having to determine the volumetric mass (except for calibration purposes), in particular without having to tediously collect intact soil cores, represents noticeable progress and should help getting much more extensive information on SOC stocks.

Soil organic carbon is the largest carbon pool in terrestrial ecosystems, thus an important component in the global carbon cycle. Moreover, because of its multifunctional role and its sensitivity to land management, SOC is one of the major indicators used for evaluating land degradation neutrality. Therefore providing guidance to help countries to estimate and monitor SOC for LDN planning is a key issue. Where SOC monitoring is necessary to verify LDN achievement, accurate data are needed (Chotte et al., 2019). DRIS has strong potential, either considering SOC concentration or stock, for supporting better evaluation of soil and land degradation, and the availability of spectrometers at increasingly affordable prices reinforces this potential.

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TABLE 1 Predictions of SOC concentration from large spectral libraries. Scanned samples were 2-mm sieved, air-dried, except for Gogé et al. (2012, 2014), who used 1-mm sieved, 55°C-dried samples, and Grinand et al. (2012) and Clairotte et al. (2016), who used 0.2-mm ground, 40°C-dried samples. Abbreviations and/or brief explanation of regression approaches: PLSR, partial least squares regression; local PLSR, uses spectral neighbours for PLSR; "double local" PLSR, uses spectral and spatial neighbours; LW PLSR, locally weighted PLSR (neighbour contributions depend on their similarity with target samples); spiking, consists of enriching the calibration set with a few target samples; MARS, multivariate adaptive regression spline; RT, regression tree; BRT, boosted RT; Cubist, rule-based RT; SVMR, support vector machine regression

Reference	Range	Library	Validation set	Approach	R ² _{val}	RPD _{val}
Shepherd & Walsh (2002)	VNIR	> 1000 topsoils, different countries	33% at random	MARS	0.80	2.2
Brown et al. (2006)	VNIR	> 4100 samples, several depths and continents	1/6th cross-validation	BRT	0.82/0.87 [†]	2.4/2.8 [†]
Viscarra Rossel & Webster (2012)	VNIR	> 10,000 samples, several depths, Australia	33% best represented	RT	0.79 [‡]	2.2 [‡]
Gogé et al. (2012)	VNIR	> 2100 topsoils representing France	33% best represented	LW PLSR	0.86	2.7
Grinand et al. (2012)	MIR	> 2100 topsoils representing France	80% at random, 10 repl.	PLSR	0.89	3.0
Gogé et al. (2014)	VNIR	> 2100 topsoils representing France	External, 24 km ² area	LW PLSR + spiking	0.58	1.4
Shi et al. (2015)	VNIR	> 2700 topsoils from China	10% well represented	PLSR	0.50	1.4
	VNIR	> 2700 topsoils from China	10% well represented	Local PLSR	0.69	1.8
	VNIR	> 2700 topsoils from China	10% well represented	"Double local" PLSR	0.74	2.0
Clairotte et al. (2016)	NIR	> 3800 top- and subsoils representing France	10% best represented	PLSR	0.85	2.0
	NIR	> 3800 top- and subsoils representing France	10% best represented	Local PLSR	0.89	3.0
	MIR	> 3800 top- and subsoils representing France	10% best represented	PLSR	0.88	2.5
	MIR	> 3800 top- and subsoils representing France	10% best represented	Local PLSR	0.92	3.4
Stevens et al. (2013)		> 19,000 topsoils representing Europe	33% best represented			
	VNIR	Cropland		SVMR	0.67/0.79 [†]	1.7/2.2 [†]
	VNIR	Grassland		SVMR	0.71/0.87 [†]	1.9/2.7 [†]
	VNIR	Woodland		SVMR	0.75/0.89 [†]	2.0/2.9 [†]
	VNIR	Other mineral soils		SVMR	0.78/0.86 [†]	2.1/2.6 [†]
Nocita et al. (2014)	VNIR	Organic soils		Cubist	0.76/0.76 [†]	2.0/2.0 [†]
		> 19,000 topsoils representing Europe	30% best represented			
	VNIR	Cropland		Local PLSR	0.79/0.84 [†]	2.1/2.5 [†]
	VNIR	Grassland		Local PLSR	0.81/0.84 [†]	2.3/2.5 [†]
	VNIR	Woodland		Local PLSR	0.79/0.85 [†]	2.1/2.5 [†]
Viscarra Rossel et al. (2016a)	VNIR	Organic soils		Local PLSR	0.76/0.76 [†]	2.0/2.0 [†]
	VNIR	> 23,000 samples, several depths, all continents	25% at random	Cubist after classif.	0.89	3.3
Lobsey et al. (2017)	VNIR	> 17,000 samples, several depths, Australia	External, two 5-km ² areas	Local PLSR	0.78-0.84	2.0

[†] Using sand as covariable. [‡] Prediction for log₁₀SOC.

TABLE 2 Predictions of SOC concentration using VNIR or MIR spectra acquired in the field, and when reported, homologous predictions using spectra acquired in laboratory conditions, on 2-mm sieved, air-dried samples (in italics; same sets and regression procedures). All studies performed PLSR (partial least squares regression) except Nawar & Mouazen (2019), who used random forest. Abbreviations and/or brief explanations of procedures: CV, cross-validation; LOO CV, leave-one-out CV; RD CV, repeated double CV; Ext., external validation with around 30% samples in general, selected either at random or on samples well represented by calibration samples (denoted "repres."; e.g. selecting every third sample after SOC-sorting); spiking, consists of enriching the calibration set with a few target samples

Reference	Range	Spectrum acquisition	No of samples and depth layers	Scale	Type of validation	R ²	RPD
Mouazen et al. (2006)	VNIR	Crushed topsoil	186, 1	Many fields, regional	LOO CV	0.76	2.0
	<i>VNIR</i>	<i>2 mm sieved, dry</i>				0.80	2.2
Stevens et al. (2008)	VNIR	From 1 m height	117, 1	10 fields in 1 region	LOO CV	0.76	2.1
	<i>VNIR</i>	<i>2 mm sieved, dry</i>				0.75	2.0
Kusumo et al. (2008)	VNIR	Intact soil core	210, 2	7 fields in 1 region	Ext. random	0.75	2.0
Morgan et al. (2009)	VNIR	Intact soil core	540, 7	6 fields in 1 region	Ext. random	0.64	1.5
	<i>VNIR</i>	<i>2 mm sieved, dry</i>				0.73	1.7
Kusumo et al. (2011)	VNIR	Intact soil core	90, 5	1 field	LOO CV	0.86	2.7
Nocita et al. (2011)	VNIR	Soil surface	113, 1	130-km transect	Ext. repres.	0.84	2.5
	<i>VNIR</i>	<i>2 mm sieved, dry</i>				0.93	3.7
Gras et al. (2014)	VNIR	Disturbed soil core	201, 1	6 fields in 1 country	CV 4 groups	0.86	2.8
	VNIR	Soil surface	201, 1	6 fields in 1 country	CV 4 groups	0.82	2.4
	VNIR	Crushed topsoil	201, 1	6 fields in 1 country	CV 4 groups	0.83	2.4
	<i>VNIR</i>	<i>2 mm sieved, dry</i>				0.80	2.3
Cambou et al. (2016)	VNIR	Disturbed soil core	288, 3	2 fields 250 km apart	Ext. repres.	0.75	2.0
Nawar & Mouazen (2018)	VNIR	15 cm depth	268+spiking, 1	4 fields in 1 region	Ext. repres.	0.78	2.1
	<i>VNIR</i>	<i>2 mm sieved, dry</i>				0.84	2.5
Veum et al. (2018)	VNIR	Borehole	708, 5	22 fields in 2 states	Ext. random	0.64	1.6
	<i>VNIR</i>	<i>2 mm sieved, dry</i>				0.82	2.3
Allory et al. (2019)	VNIR	Pit wall	132, 2 to 7	5 sites 850 km apart	Ext. repres.	0.83	2.4
	<i>VNIR</i>	<i>2 mm sieved, dry</i>				0.95	4.6
Hutengs et al. (2019)	VNIR	Soil surface	90, 1	90 fields in 1 region	RD CV	0.27	1.2
	<i>VNIR</i>	<i>2 mm sieved, dry</i>				0.66	1.3
Nawar & Mouazen (2019)	VNIR	15 cm depth	122, 1	1 field	Ext. repres.	0.65	1.7
	VNIR	15 cm depth	139, 1	1 field (another one)	Ext. repres.	0.71	1.9
	VNIR	15 cm depth	240+spiking, 1	4 fields 300 km apart	Ext. repres.	0.75	2.0
Pei et al. (2019)	VNIR	Borehole	90, 4	1 field	Ext. random	0.77	2.1
	VNIR	Borehole	58, 4	1 field (another one)	Ext. random	0.87	2.7
	VNIR	Borehole	148, 4	2 fields 3 km apart	Ext. random	0.80	2.2
Izzauralde et al. (2013)	MIR	Crushed soil	110, 3	1 field	Ext. 90% [†]	0.77	2.1
Ji et al. (2016)	MIR	5 cm depth	120, 1	1 field	LOO CV	0.86	2.6
	<i>MIR</i>	<i>2 mm sieved, dry</i>				0.90	3.2
	MIR	5 cm depth	121, 1	1 field (another one)	LOO CV	0.60	1.6
	<i>MIR</i>	<i>2 mm sieved, dry</i>				0.61	1.6
Hutengs et al. (2019)	MIR	Soil surface	90, 1	90 fields in 1 region	RD CV	0.63	1.7
	<i>MIR</i>	<i>2 mm sieved, dry</i>				0.77	2.0

[†] Calibration using some archived samples, samples from another site, and 10% samples of the current experiment

FIGURE 1 MIR and NIR spectra of SOC-rich and -poor Malagasy topsoil samples that had been 40°C oven-dried and 0.2-mm ground (59 and 5 gC kg⁻¹, respectively). Abscissas are usually wavenumbers (in cm⁻¹) for mid infrared and wavelengths (in nm) for near infrared (2500 nm = 4000 cm⁻¹). In the mid infrared, absorbance is stronger and spectra display more informative peaks (near infrared peaks mostly relate to water, e.g. at 1400, 1900 and 2200 nm)

