

Evaluating the detection limit of organic matter using point and imaging spectroscopy

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ABSTRACT

The commonly used optical technique for estimating organic matter in soils is based on a chemometric approach. Many studies have examined the correlation between spectral response and organic matter content, but none have investigated uncertainties based on unequal spectral responses in various soil types or spectral detection limit. The aim of this study was to systematically examine the spectral responses of five different soils with increasing amounts of organic matter to evaluate detection limits in point (ASD) and image (HySpex) domains. In addition, we evaluated the contribution of clay content and soils' initial organic matter on the assessed detection limit for each sensor. Large spectral variations were found between soils with the same organic matter content and between the two sensors when calculating the detection limit. Thus, applying a generic prediction model of organic matter using all soil types results in a rough estimation due to the spectrum affiliation with a certain class and not due to correct organic matter analysis. The importance of this study lies in showing that the use of spectroscopy for spectral-based organic matter detection should be practiced with caution, by highlighting the variant and inconsistent effects of organic matter in soils, and discussing the problematic assessment of organic matter when using a chemometric approach.

1. Introduction

Soil organic matter (SOM) includes all living and dead organisms within the soil matrix. It is responsible for supplying nutrients to vegetation and generating aggregates by binding soil particles together (Ashman and Puri, 2013), and it is one of the five state factors in soil formation (Buol et al., 2011). In addition, it influences other soil properties, such as compaction, water retention and soil-structure stability (Ben-Dor et al., 2009; Stevens et al., 2008), darkens soil color (Chomchan et al., 1979), and is one of the main indicators for soil-quality assessment (Parr et al., 1992; Reeves, 1997; Visser and Parkinson, 1992). SOM is one of the key criteria for the histic, folistic, mollic and umbric epipedons in the United States Department of Agriculture (USDA) classification (Bockheim, 2014a, 2014b; Soil Survey Staff, 2011) and as a diagnostic horizon in the World Reference Base (WRB) system (IUSS Working Group, 2015); it is also used to differentiate organic soils (e.g. histosols) from mineral soils (e.g. vertisols) (Buol et al., 2011), as it can range from < 0.1% in desert soils to close to 100% in organic soils (Schnitzer and Khan, 1975). Most of the soils in Israel contain low organic matter (OM) content, although some

soils, such as Terra Rosa and Mediterranean brown forest soils (see Table 1 for the equivalent WRB name), may contain higher levels of OM (Ravikovitch, 1992). Several traditional laboratory analyses exist for SOM assessment: oxidation of OM with hydrogen peroxide (Gallardo et al., 1987), the Walkley–Black titration method in which OM is oxidized with potassium dichromate in sulfuric acid (Nelson and Sommers, 1982; Walkley and Black, 1934), and the loss-on-ignition (LOI) method (Ben-Dor and Banin, 1989). However, these methods are either expensive or time-consuming.

In the past few decades, there have been many studies devoted to investigating the correlation between spectral data in the visible, near-infrared and shortwave-infrared (VNIR–SWIR) regions (400–2500 nm) and OM content or its correlated attribute organic carbon using the spectroscopy and imaging spectroscopy domains (Ben-Dor and Banin, 1995; Ben-Dor et al., 1997; Ben Dor et al., 1999; Daniel et al., 2003; Hummel et al., 2001; Jin et al., 2016; Sudduth and Hummel, 1993; Krishnan et al., 1980; Lamsal, 2009; Liu et al., 2008; Peón et al., 2017; Shi et al., 2015; Tian et al., 2013; Volkan Bilgili et al., 2010; Wang et al., 2016). Although many studies use the entire spectral range, some have found specific spectral ranges for chemometric modeling, mostly

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Table 1

The soil types of the Israeli soil classification system with the equivalent World Reference Base (WRB) system, their texture and OM properties

Code	Soil series Israel	WRB	Clay (%)	Silt (%)	Sand (%)	OM _S (% wt)
H	Hamra (brown-red sandy soils)	Rhodic Luvisols	0	0	100	0.61
L	Loess	Luvic Calcisols	9.8	43.6	46.6	4.13
R	Rendzina	Calcic Leptosols	14.5	53.5	32.0	5.23
T	Terra Rosa	Epileptic Luvisols	17.1	59.9	23.0	9.01
S	Sand (sand dunes)	Arenosols	0	0	100	0.27
	Compost	–	–	–	–	35.8

from 400 to 1000 nm (Cozzolino and Morón, 2006; Henderson et al., 1992; Krishnan et al., 1980; Tian et al., 2013; Vaudour et al., 2016). Numerous studies have examined the linear fitting between OM and spectra, but none has investigated how different contents of OM affect the prediction model in different soil types, nor have any taken into consideration the detection limit (DL) of the spectral-based approach to determine OM content with reliable results. The DL, or limit of detection (LoD), is defined as the lowest detectable amount of analyte in a sample in a given method (Broughton and Ermer, 2005). It is expressed as the analyte concentration corresponding to the sample < blank > + 3 standard deviations (σ) (Shrivastava and Gupta, 2011).

Since different soils exhibit different spectral responses due to their chemical and physical compositions, the effect of the same OM content is assumed to be variant and inconsistent. As a result, a chemometric approach to evaluating SOM cannot be successful using generic statistical approaches, and the DL of OM for every soil type must be accurate.

In this study, five different soil types from different locations in Israel were collected. Each soil was air-dried, ground and sieved to 2 mm and then was divided into 18 sub-samples which contain varying percentages of OM (an industrial compost—used as an indicator of OM content). The compost was added in small intervals of 0.2% until it reached a total of 2%, which is sufficient for identifying changes in each individual spectrum. In order to achieve commonality and compare between soils with low OM (Sand and Hamra) and soils with high OM (Terra Rosa) in a joint model, six additional sub-samples with higher percentages of compost were added (2.5, 3, 5, 11.1, 20, 33.3 and 42.9%). The main objective of this study was to systematically examine the effect of varying amounts of a known OM contents to evaluate the soil spectral detection limit (SSDL), using both point and imaging spectroscopy under controlled laboratory conditions.

2. Materials and methods

2.1. Soil samples and laboratory measurements

Five different soil types were collected in Israel from the surface level. In addition, an industrial compost material was used as an OM demonstrator source which is added to the initial OM content. All samples were air-dried, ground and sieved to 2 mm. The soils' texture was measured with a laser diffraction particle-size analyzer (Mastersizer 2000, www.marlven.com) and the natural (initial) SOM content (OM_S) was measured by LOI (400 °C for 8 h) method (Ben-Dor and Banin, 1989; Davies, 1974). Table 1 provides the clay, silt and sand distribution and OM_S measured by the LOI method for each soil type.

2.2. Sample preparation

Each soil was divided into 18 sub-samples of 30 g each with different percentages of added compost. Distilled water was added to the

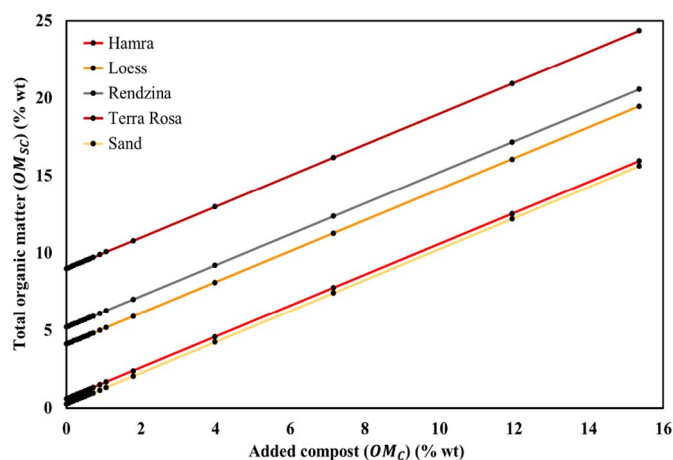


Fig. 1. The added compost (OM_C), the initial OM content in soil (OM_S) (marked as the first sample in each series) and the total OM content in the soil samples (OM_{Sc}).

sample until saturation, to enable better soil–OM mixture, stirred and then air-dried and mashed to 2 mm. Fig. 1 presents the percentage of the added compost (OM_C), the initial organic matter in the soils (OM_S) (where OM_C = 0), and the total organic matter content (OM_{Sc}) originated from both the soil and the compost for each soil type. For weight measurements, we used an Electronic Semi-Microbalance R200D (www.sartorius.com) with standard deviation of less than ± 0.00002 g.

2.3. Sensor configuration and spectral data correction

The spectrum of each soil sample was measured using both point and image spectrometers using the standards and protocols proposed by Ben Dor et al. (2015). The point spectrometer was the portable Analytical Spectral Devices spectrometer ASD FieldSpec in the Remote-Sensing Laboratory at Tel Aviv University, Israel. The spectrometer has three detectors with 1 nm interpolated spectral resolution that supply 2151 bands in the VNIR (350–1000 nm), SWIR1 (1000–1800 nm) and SWIR2 (1800–2500 nm) regions.

The hyperspectral images were acquired at the Remote-Sensing Laboratory of GFZ German Research Centre For Geosciences, Potsdam, Germany, using the HySpex imaging spectrometer. The sensor contains two detectors in the VNIR range and SWIR range with spectral resolution of 3.6 and 6 nm, respectively. The integration time was adjusted manually so that the maximum radiance within all samples could be covered without saturation. Subsequently, the total 436 bands (between 400 and 2500 nm) were reduced to 399 bands due to extraction of overlapping wavelengths. Additional information regarding the soil measurement and spectral correction procedures can be found in Rogass et al. (2017).

Fig. 2 shows the HySpex images together with the spatial arrangement of the samples. The orange circles mark the internal soil standard as proposed by Ben Dor et al. (2015) and the yellow circle marks the compost sample.

Following the initial preparation, samples were measured using a contact probe equipped with a halogen bulb and Spectralon® as a white reference and corrected using the internal soil standard as discussed in Ben Dor et al. (2015). Due to data acquisition differences between the sensors (point vs. pixels), different approach for data collection needed to be taken. Using the ASD, each soil type was measured 3 times (to get the average spectrum of the sample) while first sample (compost-free sample or blank) of each soil type was measured 20 times to get the mean and standard deviation for a later DL calculation. However, the image acquisition was performed only once, so in order to obtain the 20 spectra of the blank sample, the average spectrum and the standard deviation were calculated from 20 regions of interest (ROI) of 5 pixels each which resemble 20 independent measurements. The average

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