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Reflectance measurements of soils in the laboratory: Standards and protocols

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ABSTRACT

For the past 20 years, soil reflectance measurement in the laboratory has been a common and extensively used procedure. Based on soil spectroscopy, a proxy strategy using a chemometrics approach has been developed for soils, along with massive construction of soil spectral libraries worldwide. Surprisingly however, there are no agreed-upon standards or protocols for reliable reflectance measurements in the laboratory and field. Consequently, almost every user reconstructs his or her own protocol based on the literature, experience, convenience and infrastructure. This yields significant problems for comparing and sharing soil spectral data between users, as spectral variations can be encountered from one protocol to the next. This further prevents the generation of a robust model for a given soil property using the worldwide data archive. To solve this problem in the laboratory, a joint project between CSIRO - Perth and Tel Aviv University (TAU) was conducted to establish a standard protocol for soil measurement, along with the use of an internal standard procedure to generate correction factors to normalize all possible variations to a soil benchmark (SBM) setup. The method is based on a proof of concept reported by Pimestein et al. (2011) for the use of an internal soil standard (ISS), a concept adopted from the wet chemistry discipline. To formulate the ISS method proposed herein, we used samples from two sand dunes that were characterized for their mineralogy, stability and reflectance reproducibility to be suitable for the standardization mission. These samples were found to be stable in space and time and to hold a stable soil structure and spectral response common to soils. Five soil samples were used to examine the performance of the suggested ISS approach. The ISS and soil samples were carefully measured for reflectance at the CSIRO laboratory using a new calibrated ASD and different protocols and procedures harboring minor and major differences. In addition, the exact same ISS and soil samples were measured in the TAU laboratory with a different ASD, protocols and ambient conditions. A systemic quantitative study was conducted to judge the performance of the ISS method. The results demonstrated that each protocol provides significant spectral variations from the SBM protocol, but all can be corrected to the SBM measurements. The results are promising as they can be achieved with any protocol if the systematic variations are kept constant. The ISS samples can also be used to track spectrometer deterioration and measurement-setup stability. The current paper provides information on how to obtain and use the ISS, and recommends a simple protocol for measuring soil reflectance under laboratory conditions.

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1. Introduction

In the last decade, soil reflectance has become a well-recognized tool to assess soil attributes rapidly and accurately in the laboratory domain (Ben Dor, 2010). Soil proxies for many attributes, soil-mineral recognition and soil-type discrimination are the major applications that soil reflectance can contribute to soil practices. In recent years, it has been shown that soil reflectance can be used from both field and air domains using two major spectral technologies: portable point and airborne imaging spectrometry (Ben-Dor et al., 2009). With the rise in available

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portable spectrometers, more users in the soil discipline are entering the field and many soil spectral databases are being constructed in the laboratory to assist in other domains (i.e. http://www.isric.org/data/ icrafisric-spectral-library).

Whereas spectral libraries of rocks and minerals have been quite popular over the past 30 years (Clark 1999; Crowley 1991; Crowley et al., 2003; Hunt 1977,1979,1980; Hunt and Salisbury, 1970,1971,1976; Hunt et al., 1971a,1971b,1971c; Gaffey 1985,1986; Swayze et al., 2007) and can be easily shared, for soils, despite the establishment of a few soil libraries, spectral sharing is quite limited. This is mainly because in rocks and minerals, mostly the absorption positions are important; in soils, for proxy models, not only are the absorption positions important, but also the intensity and shape of the spectrum are crucial. The first soil spectral library (SSL) was published by Baumgardner et al.





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(1985), who collected hundreds of soil samples from the US and categorized them into five major soil types. Later, when soil proxy became a feasible method, it was understood that the SSL must be accompanied by chemical attributes of the soil samples (together with their spectra), and these two databases (spectral and chemical) were integral parts of the SSL (Shepherd and Walsh, 2002; Brown et al., 2006).

In the last 5 years, there has been a growing effort to generate more local SSLs by professional users who would then contribute and exchange their data internationally to form a world SSL archive (Viscarra Rossel, 2009). Generating SSLs in a large-scale domain is a growing and important mission that has recently been adopted by many organizations and entities, at both the national (e.g. Knadel et al., 2012) and continental (e.g. the LUCA SSL in Europe; Stevens et al, 2013) levels. It is predicted that there will be an increased in activity in this research as more SSLs are established and shared by the soil spectroscopy community.

To succeed with a soil proxy model, both chemical and spectral data have to be carefully acquired, to minimize the systematic and random effects. However, this is impossible when the population and measurement conditions (e.g. soil types, instrumentation and protocols) vary or are unknown (Brown, 2007). This problem generates significant constraints to obtaining a robust model for given attributes using "merged" SSL data, and blocks any attempts to compare them (Wetterlind and Stenberg, 2010). In contrast to chemical analyses, there are no agreedupon protocols for soil spectral measurements. Users therefore prefer to keep their successful protocol active and are not open to examining others. One way to prevent possible systematic variations might be achieved mathematically, by following procedures such as spectral derivation or standard normal variate (SNV). However, in the case of high variations, these procedures could be problematic. A more basic method that deals with the physical parameters and spectral origin is thus needed.

To solve this problem, Pimstein et al. (2011) suggested adopting the idea of internal standards from the wet chemistry discipline for soils. Those authors demonstrated that well-known and agreed-upon reference material that is measured under any setup in any laboratory can be used to align one laboratory's spectral measurements to another's. They termed this material internal soil standard (ISS); their sample was not the ideal example to be shared throughout the soil spectral community; rather, it was useful as a proof of concept. Accordingly, the search for an ideal ISS remains active and the ISS idea cannot yet be implemented in SSLs worldwide.

This paper reports on a comprehensive study conducted to seek, identify and establish an ideal ISS sample and scale up Pimstein et al.'s concept for practical use. This is in order to create standards and protocols that will enable the comparison and use of every soil spectral measurement that has been acquired by different spectrometers, geometries and external conditions. The ISS sample was examined under minor and extreme conditions to check its suitability as a robust standard and to develop an agreed-upon spectral measurement for the soil spectroscopy community. This study involved a comprehensive collaboration between Tel Aviv University (TAU), Israel, and CSIRO – Perth, Western Australia.

2. Theoretical background

2.1. Factors affecting soil spectra

Two main factors can affect soil spectra: nonsystematic and systematic. The nonsystematic effects are those arising from uncontrollable phenomena, such as random noise and uncertain effects and instabilities (noise). To minimize the nonsystematic "noise" effects, it is important to maintain a consistent protocol. To achieve this, users must keep the instrumentation factors (instability of the spectrometer, illumination source, and detector output), as well as sample preparation, constant using an agreed-upon protocol. If these factors are not controlled, noisy and inconsistent soil spectra may result. The systematic effects are those factors that arise from controlled responses that change from one instrument to the next but stay constant in a selected protocol. To prevent systematic effects, controllable and recordable factors such as the white reference (WR) sample, spectral configuration, measurement geometry, fore optic status, operator, particle size distribution and environmental conditions, are kept constant or tracked. If these factors are not controlled, the results may vary and hinder the ability to obtain good results from the chemometric analysis while using multisource SSLs; in practice, it may also prevent the sharing of SSLs between potential users.

Whereas the nonsystematic effects can be minimized by using an agreed-upon protocol, the systematic effects can vary from one laboratory to another. Consequently, a method to minimize the systematic effects is strongly required and effort in establishing measures to align different SSLs that have been generated using different protocols needs to be made. This issue is doubly important as such a method is lacking and cross-calibration between laboratory infrastructures is uncommon or nonexistent.

2.2. Description of the ISS principle and method

As previously discussed, the internal standard idea was adopted from the wet chemistry discipline to minimize systematic effects, where an agreed-upon and well-known (species and concentration) material is used to align the readings of any method (Willis, 1972). The internal standard idea is based on the fact that the nonsystematic effects are minimized by the protocol used in the professional laboratory, while the different systematic effects between protocols are corrected for using the alignment factor generated from the standard's reading. In general, an ideal ISS should be inexpensive, simple to use, easily delivered overseas, homogeneous, stable in space and time, and useful for both radiometric and spectral calibration. Pimstein et al. (2011) concluded that the internal standard also has to be as similar as possible to soil grain size (shape, size and nature) and if possible, it should have stable (and preferably chemically featureless) spectral performance across the entire spectral region. They indicated that their sand dune sample (90% quartz), which underwent a bleaching process with HCl and the DCB method (Mehra and Jackson, 1960) to remove calcite and free iron oxides, respectively, and was characterized by an average grain size fraction of <2 mm, is the preferred ISS sample. It was shown that other materials (flat polyethylene surface and glass crushed to <2 mm) cannot perform the correction as effectively as the sand sample and concluded, as previously mentioned, that the ISS must have "soil characteristics" in terms of shape and nature. Nonetheless, based on their proof of concept, they strongly recommended finding a better ISS sample from natural sources that satisfies their previous suggestions and does not need to be subjected to chemical processes. They also pointed out that such a sample must be tested, and be easy to disseminate to the scientific community at low (or no) cost with long-term availability to all.

3. Material and methods

3.1. ISS and soil samples

3.1.1. Internal soil standard (ISS)

We devoted an international effort to locate the ideal ISS samples that would satisfy Pimstein et al.'s (2011) criteria. Recently, in Australia, two sites were characterized as bright, homogeneous sand dunes (Fig. 1). They were found along the coastline of Wylie Bay (WB) and Lucky Bay (LB) in southwestern Australia. The sand dunes were sampled by collecting the top 10 mm (about 50 kg each) and brought to the laboratory to be further inspected for their fulfillment of ISS criteria. In WB 2 (33°49′ 23.89″S 121°59′51.81), the sand was sampled on a moderate slope of a 30-m high dune hill (under dry conditions), and in LB (33°59′ 17.22″S 122°13′51.89″), it was sampled at the waterfront (wet conditions). After arrival at the laboratory (2 days after sampling), the sand

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