

VARIATION AND STABILITY OF SOIL REFLECTANCE MEASUREMENTS WITH DIFFERENT ASD SPECTROMETERS UNDER DIFFERENT CONDITIONS

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Abstract

This paper summarizes a comprehensive and systematic study performed to estimate and assess the possible variation in soil reflectance using different spectrometers and measurement methods. A group of twelve chemically and physically characterized soils were measured spectrally by multiple operators using three different ASD spectrometers with different protocols and measurement conditions. Three different "non-soil" materials were also used to examine the stability of the sensors with known samples that are assumed to be unaffected by external conditions. Lower levels of variability between instruments ($SD\ 0.014 \pm 0.01$ vs. 0.027 ± 0.01) were observed among the analyzed soils when one specific protocol was applied by the same operator, in comparison to operation of the instruments by different users under different protocols. The variability between replicates (three to five) collected for each sample was significantly lower when different operators performed the measurements using a common measurement protocol. We propose that a measurement protocol for soil reflectance be established and agreed upon by all users worldwide to better exchange soil spectral libraries and compare quantitative analyses. In addition, there is a strong need in the soil spectral community for a method to correct for unexpected variations. We set the requirements for such an approach to enable the variable spectra of a given soil sample to be translated into a common denominator.

Introduction

Spectroscopy is considered to be a solid and fully developed technique for qualitative and quantitative analysis of different materials and targets. Evaluations of vegetation, water, soil and food materials are among the common applications for which spectral information across the VIS-NIR-SWIR spectral range is used for rapid chemical analyses (Workman and Shenk, 2004). Many applications have been developed for soil spectroscopy over the past 20 years, which have propelled the soil sciences forward by providing a rapid and accurate perspective for quantitative analyses of soil, skipping the traditional "wet" laboratory work (Ben Dor et al., 2008; Malley et al., 2004). Accordingly, portable spectrometers are being developed and utilized worldwide to characterize and monitor natural resources such as soils, rocks, vegetation and water. In addition, a wide range of soil spectral measurements are being gathered around the globe with the intention of building a universal soil spectral library (Viscarra Rossel, 2009). Considering that the spectral performance of spectrometers may vary among model types, or even among series of a particular model, it is important to characterize these differences, investigate the source of the errors and establish a protocol for the optimal performance of soil spectral measurements to enable adequate comparisons of spectra (Price, 1994). Spectral differences in the same material have already been identified in other disciplines. Williams and Krischenko (1986) showed that the same wheat population measured by two spectrometers of the same type and brand yields different spectral models to predict protein content. Although both models were relevant for practical applications, the spectral prediction model developed with data from one spectrometer could not be used with data from the second spectrometer. Sohn et al. (2008) studied the quantitative performance of different spectrometers using NIRS technology that analyzed whole barley grains, and found different

accuracy levels in predicting some of the analyzed components. To compensate this reported variability, over the last 20 years several algorithms have been proposed for spectral standardization, but these have not yet been largely adopted by the soil and rock sciences community due to their strong degree of complexity.

Unlike spectra of rocks and minerals, which are easily recognized for diagnostic assessment by specific and significant absorption features, some of the important spectral features used for soil models are weak and sensitive to measurement conditions. Consequently, technical factors that alter the reflectance spectrum during its collection need to be evaluated and studied to eliminate as many of them as possible. The need to minimize such factors in a robust way is important, especially today, when many new users are entering the field of soil spectroscopy and large amounts of data are being continuously collected and exchanged worldwide. As a result, strict protocols should be included in these measurements so as to make the most productive and efficient use of these invested resources.

The purpose of this study was therefore to compare three spectrometers from the same vendor and model while using a standard protocol, as well as to compare other users' protocols using the same population of soil types in different locations and under different environmental conditions. Another goal of this study was to develop a practical way of minimizing uncontrolled spectral variation by testing different measurement protocols.

Materials and methods

Three FieldSpec spectrometers from Analytical Spectral Devices, Inc. (ASD) that belong to different remote-sensing laboratories in Israel were selected for this study. One of them was brand new (SAHAF), while the others (TAU and BOKER) were each more than 6 years old. The three instruments were calibrated at ASD's laboratories (Boulder, CO) a maximum of 3 years before the current measurements. The instruments were furnished with a 1- to 2-m-long fiber optic cable with a foreoptic of 25° field of view, which was coupled to an ASD High-Intensity Contact Probe powered by a stabilized tungsten-halogen lamp. The measurements were optimized and calibrated to spectral reflectance using a Spectralon standard white reference panel (Halon, Labsphere, Inc., North Sutton, NH). Twelve soil samples were selected from the Israeli Soils Library (<http://www.tau.ac.il/~rslweb/slis.html>) for use in this study. The basic criteria for selecting the soil types were to include large albedo variability and different absorption features of well-known chromophores, i.e. clay/sand ratio, CaCO₃, Fe-oxides, and organic matter. Each of the soil samples was subjected to a careful wet chemical analysis as traditionally conducted in soil sciences and as previously described in detail by Ben-Dor and Banin (1995). This included drying and grinding the soil to less than 2 mm and aggregate analysis of the <2 mm fraction. In addition to the soils, we also included internal standard materials that are spectrally featureless across the entire spectral region, with a grain size similar to that of the soil material, and which provide a spectral response that is inherently independent of external conditions (temperature, humidity, soil sample smoothness). Three materials were selected as internal standards: 1) a gray mat polyethylene panel (*Formica*)—a 20 x 20 cm panel weighing 10 g with a uniform and homogeneous surface, 2) crystal clear glass (*Glass*) which was crushed to pass a 3-mm sieve, and 3) bleached dune sand (*Sand*)—a natural sand material (<2 mm) with 90% quartz content. The *Sand* material used for this study was subjected to a bleaching process by chemically removing carbonates using dilute HCl solution (Jackson, 1967) and free Fe-oxides by the dithionite-citrate-bicarbonate method (Mehra and Jackson, 1958).

Two experiments were carried out. In the first (Exp. 1), the soil spectral measurements were performed simultaneously on the three different ASD spectrometers using a strict measurement protocol developed at TAU. In general, this protocol provides timing instructions for operating the equipment and for the measurement sequences, as well as measurement configuration and data quality control. The *TAU Protocol* for measurements with the ASD high-intensity contact probe is detailed as follows:

- 1) Warm up the spectrometer for at least 30 min before starting the measurements (the vendor recommends 1 h).
- 2) Configure the instrument for collecting 40 samples as Spectrum, Dark Current and White Reference measurements.
- 3) Clean the contact-probe glass and check that the fiber foreoptic is in position.
- 4) Gently connect the fiber optic to the contact probe, taking care not to overly tighten the connection, and turn on the bulb for 15 min before starting the measurements so that the lamp temperature is stabilized.
- 5) For laboratory measurements, attach the contact probe to a mechanical pole. This stabilization step eliminates probe shake during the measurements.
- 6) Optimize the spectrometer using a Spectralon standard white reference panel (Halon) every 20 measurements.
- 7) Calibrate to spectral reflectance using the Halon white reference plate every 10 measurements (not necessary if radiance is being measured).
- 8) Collect a reflectance (or radiance) measurement of the internal standard being used.
- 9) For each sample, place soil material (>2 mm) in the selected container (e.g. transparent plastic bins) and smooth it as possible.
- 10) Measure the reflectance (or radiance) of the sample.
- 11) Mix and smooth the surface of the soil sample as much possible with the aid of a knife or spatula.
- 12) Repeat the set of measurements (steps 9 and 10 above) three to five times (replicates), including steps 6 to 9.
- 13) Check the standard deviation of each sample. If it exceeds 2% then go to step 6 and repeat the measurements.

The second experiment (Exp. 2) was performed with the same soil samples and standards in each laboratory with the spectrometer under study (TAU, SAHAF, BOKER), using different operators, contact probes and their own in-house protocol (differing mostly white reference calibration, soil preparation and contact-probe stabilization). Whereas the measurements in Exp. 1 were performed on the same day and time under the same conditions, Exp. 2 was performed in different geographical locations, on different dates and with different protocols and users. For both experiments, the measured soil samples varied between 15 and 20 g and were air-dried. The measurements of the internal standards (*Formica*, *Sand* and *Glass*) were taken following each collection of white references. In each experiment, three to five replicates were taken for each sample and their averages were used for further analyses. The soils were gently stirred between measurements and the surface was smoothed as in step 9 of the *TAU Protocol*.

Results and Discussion

Albedo-intensity variability

In Exp. 1, the spectral variability of the analyzed soils fluctuated among the instruments from a mean spectral standard deviation of 0.003 (CV 0.95%) for soil K2 (sandy clay loam—Rendezina soils of the valley) to 0.024 (CV 8.7%) for soil B (Mediterranean brown forest soil). Figure 1 illustrates the mean spectra and standard deviations of soil K2 for Exps. 1 and 2. As can be seen from Figure 1b (Exp. 2), aside from the variability in albedo intensity, significant modifications in the shape and location of the absorption features can be distinguished in the SWIR region, especially in the BOKER case. The differences between the results of Exps. 1 (low CV) and 2 (high CV) suggest three error sources: 1) variation in the spectral assemblies (spectrometer and its accessories, e.g. different contact probes), 2) different protocols/operators, and 3) the surrounding conditions (including sample preparation). Spectrometer variation can be ascribed to malfunction in the use of the contact probe (e.g. unstable fiber optic installation or shaking during the measurement). One clear example of the protocol/operator source of variation corresponds to the approach used for holding the contact probe (i.e. using bare hands or holding it with a mechanical pole). Finally, the surrounding measurement conditions can vary according to the humidity, which affects the adsorbed water molecules. Relative humidity is known to affect the absorption features of water at 1400 and 1900 nm (Ben Dor et al., 1998). The BOKER climate is arid and the relative humidity is lower than that at TAU and SAHAF (49%, 76% and 72%, respectively). This suggests that in Exp. 2, the variation stems from a combination of the above three factors whereas in Exp. 1, factor 1 is dominant.

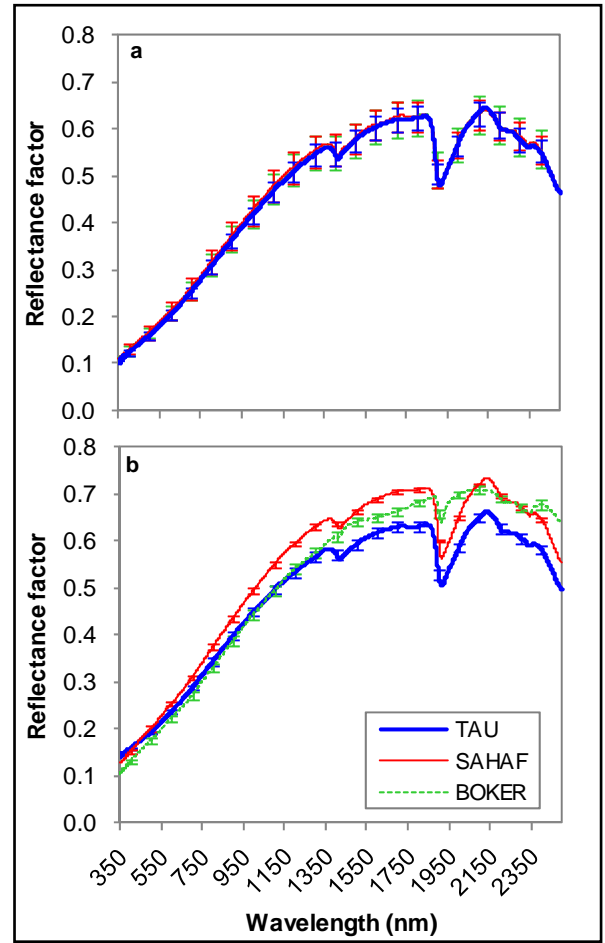


Figure 1. K2 reflectance from Exp. 1 (a) and Exp. 2 (b). Error bars represent two standard deviations between replications

The soil variability between instruments was calculated for each experiment according to:

$$S\sigma = \frac{\sum_{\lambda=350}^{2500} \sigma(TAU\rho_{\lambda}, SAHAF\rho_{\lambda}, BOKER\rho_{\lambda})}{2151} \quad (1)$$

where $S\sigma$ is the mean spectral standard deviation, σ is the standard deviation function and ρ_{λ} is the mean reflectance per wavelength (λ) for each instrument. As can be seen from Table 1, 9 out of 12 soils showed higher variability when measured by different operators under different conditions, using different measurement protocols (Exp. 2). It is assumed that these variations are a result of both

instrument and operator stability, and of the variation in the soil surface exposed to the radiation (each soil was measured in three to five replicates that were prepared for measurement by re-mixing the soil prior to each measurement).

Table 1. Mean spectral standard deviation between instruments, by soil and experiments

Soils	Exp. 1	Exp. 2	Soils (cont.)	Exp. 1	Exp. 2
A1	0.0033	0.0083	E2	0.0101	0.0326
A2	0.0066	0.0142	K1	0.0399	0.0380
B	0.0238	0.0150	S	0.0122	0.0445
C	0.0139	0.0363	K2	0.0033	0.0342
E1	0.0242	0.0220	SHo	0.0098	0.0340
EC	0.0119	0.0169	E3	0.0100	0.0313

Similar results were obtained for the references when analyzing the differences between the instruments. Figure 2 shows the average spectra and between-instrument standard deviation of the analyzed internal standards for Exps. 1 and 2. As can be seen, *Formica* has some spectral features in the VNIR and SWIR part of the spectrum that are suitable for assessing the spectral performance of the spectrometer in question. The *Glass* can be characterized as a featureless material for which no significant spectral response can be seen and the *Sand* spectrum is characterized by weak but pronounced absorption features of hygroscopic water at around 1400 and 1900 nm, kaolinite at 2200 nm and some leftover features of Fe-oxide minerals at 550 nm.

Based on the previously defined criteria, these few and relatively weak absorption features plus the high amount of spectrally featureless (quartz) content in both *Glass* and *Sand* make them theoretically optimal for spectral stability assessment (radiometric response). In addition, the *Formica* plate, which holds specific stable absorption-spectral features, can be used as a standard to assess the spectral stability of the spectrometer in question (spectral response). Moreover, a comparison of the two experiments confirms the larger observed variability between the instruments in Exp. 2, whose measurements were performed in different locations under different measurement protocols. Particularly interesting is the fact that in comparison to the variability in *Sand* vs. *Glass*, *Formica* was relatively stable between both

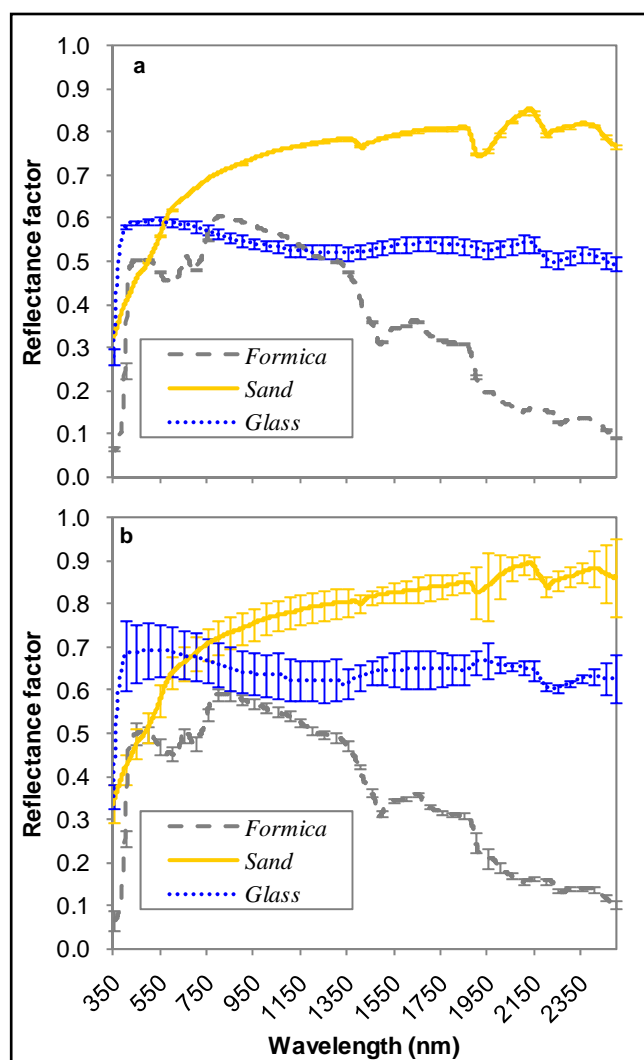


Figure 2. Internal standards' reflectance from Exp. 1(a) and Exp. 2 (b). Error bars represent two standard deviations between instruments

experiments. This indicates that inert and constant surface materials are less affected by the measurement conditions (no water adsorbed on the surface and the sample surface shape is constant).

Aside from the variability between instruments, it is important to characterize the variability between replicates for each instrument. For each soil, the standard deviation between replicates in Exp. 1 was related that obtained in Exp. 2 ($SD_{Exp. 2}/SD_{Exp. 1}$). Table 2 shows the statistics of the deviation from unity of this relationship (absolute values). BOKER showed a significantly higher deviation from unity than the other locations in overall mean, minimum and maximum values. This means that the standard deviation between replicates of Exp. 2 was much higher than that obtained during Exp. 1, which creates the basis for possible significant differences in average spectra. The differences between TAU and SAHAF are more subtle but nevertheless, in general, the results show TAU variability between replicates among both experiments (i.e. lower mean and higher maximum values).

Table 2. Statistics of the deviation from unity of the relationship between Exp. 1 and Exp. 2 of the soils' standard deviation between replications ($n = 12$). Q1: 1st quartile, Q3: 3rd quartile

Statistic	TAU	SAHAF	BOKER
Mean	0.515	0.727	2.173
Min	0.032	0.015	0.156
Q1	0.326	0.367	0.311
Q3	0.604	0.770	1.066
Max	1.317	3.007	12.312

As already mentioned, during Exp. 1 the three instruments were used under the *TAU Protocol* and operated by the same person. In contrast, during Exp. 2, the three instruments were operated by different operators who applied different measurement protocols. Therefore, by comparing the measurements of each instrument between experiments, the variability associated with the measurement protocol and with the operator factor can be estimated. As TAU measurements in both experiments were based on the *TAU Protocol*, this comparison represents only the operator factor.

Table 3. Statistics of the spectral mean difference between Exp. 1 and Exp. 2 ($n = 12$). Q1: 1st quartile, Q3: 3rd quartile

Statistic	TAU	SAHAF	BOKER
Mean	0.015	0.046	0.032
SD	0.009	0.026	0.027
Min	0.003	0.009	0.007
Q1	0.010	0.027	0.017
Q3	0.017	0.063	0.034
Max	0.032	0.085	0.109

The significantly lower differences between the TAU measurements relative to the other two instruments confirms that the differences between spectral measurements obtained by different operators are reduced when using a common measurement protocol (e.g. *TAU Protocol*). Specifically, the advantages of this protocol are based on the use of a mechanical pole to hold the contact probe, which reduces vibrations associated with hand-holding the probe.

Summary and Conclusions

Two experiments were conducted within the framework of this research to compare three ASD spectrometers, three measuring conditions (spectroscopy measurement protocols) and three materials considered to be internal standards for soil spectral measurements. When comparing the variability among replicates of a particular soil type, soil granulometry was seen to largely affect this variability. In Exp. 1, in which only one operator applied the *TAU Protocol* for all three instruments, the mean coefficient of variation (between replicates) among the spectra collected with the most recently calibrated instrument ranged from 0.95% to 8.70%. This variability represents the source of error, generated by the soil characteristics themselves, which is followed by the variability obtained between instruments. In general, this variability was significantly ($p < 0.05$) higher when the soil samples and references were measured by different operators using different protocols. Finally, by analyzing the variability between replicates for each instrument in each experiment, it was possible to differentiate the operator/protocol effect on the overall variability. As the measurements performed with the TAU instrument were collected by different operators but using the same protocol, the differences between Exps. 1 and 2 for each instrument showed that when applying this protocol, significantly lower differences are obtained than when applying a non-common protocol. Therefore, the measuring protocol to be used in soil spectral measurements is as important as the instrument, and it is recommended that the *TAU Protocol* be applied, as it enables comparing spectra collected by different operators. For those cases involving changes in instrument or location, following the same protocol may not be enough. Therefore, it is suggested that a simple and practical standardization methodology be developed to neutralize those effects that were observed in this work to be crucial.

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