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Application of mid-infrared spectroscopy for rapid characterization of key soil properties for engineering land use

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Abstract

Methods for rapid and accurate soil tests are needed for the index properties of material attributes commonly applied in civil engineering. We tested the application of mid-infrared (MIR) spectroscopy for the rapid characterization of selected key stability-related soil properties. Two sample sets, representing different soils from across Lake Victoria basin in Kenya, were used for the study: A model calibration set ($n=135$) was obtained following a conditioned Latin hypercube sampling, and a validation set ($n=120$) was obtained from independent sites using a spatially stratified random sampling strategy. Air-dried ground (<0.5 mm) soil was scanned using a high-throughput screening accessory for diffuse reflectance attached to a Fourier transform infrared spectrometer. The soil properties were calibrated to smoothed first derivative MIR spectra using partial least-square regression (PLS), and screening tests were developed for various limitation classes applicable in civil works using the soft independent modeling of class analogy (SIMCA). The hold-out full cross-validation coefficient of determination (r^2) ≥ 0.8 was obtained for the liquid limit (LL), linear shrinkage (LS), coefficient of linear extensibility (COLE), air-dried moisture content, (**W**) and cation exchange capacity (CEC). Further independent validation gave $r^2 \geq 0.73$ and the ratio of prediction deviation (RPD) 4.4–2.1 for LL, LS, COLE, W, CEC, plastic limit (PL), plasticity index (PI), and volumetric shrinkage (VS). The independent validation likelihood ratios for the diagnostic screening tests were: LL > 55%, 4.2; PI > 30%, 2.7; LS > 12%, 2.4; exchangeable sodium (eNa) > 2 cmol (+) kg^{-1}, 2.3; exchangeable sodium percent (ESP) > 10%, 1.8; **W** > 8.3%, 1.6, and Activity number (A) > 1.25 units, 1.5. MIR can provide the rapid assessment of several soil properties that yield stability indices in material testing for engineering land use. Further studies should test the ability of MIR PLS for establishing broader calibrations across more diverse soil types and the direct correlation of MIR to material functional attributes.
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Keywords: Mid-infrared spectroscopy; Soil properties; Mixed and separate depth models; Engineering land use; Diagnostic spectral screening

1. Introduction

The increasing demand for quality-intensive soil information, for environmental monitoring, modeling, and civil engineering, calls for the application of the most cost-effective methods of soil data acquisition (Shepherd and Walsh, 2007). Conventional laboratory methods for determining soil properties are often not cost effective as they require a different test for each property and a wide range of equipment and procedures. In addition the methods are often

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susceptible to high measurement errors and low precision (Cantarella et al., 2006; Viscarra Rossel and McBratney, 1998). Infrared spectroscopy (IR) has demonstrated several advantages over wet chemistry laboratory methods: (i) IR is rapid (sample preparation and spectral measurement is achieved within 2 min allowing a high throughput of 200–400 samples per day); (ii) a single spectrum integrates information on a number of soil properties, and; (iii) IR measurements are highly precise (Linker, 2012; Shepherd and Walsh, 2002; Viscarra Rossel et al., 2006). For example, Shepherd and Walsh (2003) showed how IR may be used to help improve the accuracy of the reference wet chemistry method. Shepherd et al. (2005) demonstrated that IR was more repeatable than wet chemistry methods by halving the measurement standard deviation (SD). Howari et al. (2002) found a SD of 0.8×10^{-2} nm of spectra readings from soil evaporates and salt crusts, whereas corresponding SD from wet chemistry was 0.32. These properties make spectroscopic analyses combined with multivariate calibration attractive for environmental monitoring and modeling, precision agriculture, and civil engineering (Shepherd and Walsh, 2007; Viscarra Rossel et al., 2008).

The use of diffuse reflectance mid-infrared spectra (MIR) (2500–25000 nm), in combination with partial least-square regression (MIR PLSR), has been investigated in numerous soil studies (Linker, 2012; Shepherd and Walsh, 2007; Viscarra Rossel et al., 2006). However, no studies are available on the application of MIR PLS soil analyses to materials being tested for civil engineering applications. Waruru et al. (2014) demonstrated the satisfactory to weak performance of near-infrared (NIR) (700–2500 nm) for the estimation of soil engineering properties, whereas MIR is reported to be more resourceful than NIR for the prediction of several soil properties (Viscarra Rossel et al., 2006).The question of whether mixed depth (set of samples extracted from different depth intervals) or separate depth (set of samples extracted from one depth interval) MIR PLS models are more effective for the characterization of soil properties has not yet been explored. Few attempts using visible–NIR spectral region (400–2500 nm) (Nanni and Demattê, 2006) have been found, indicating the significant differences in the performance of surface and subsurface sample datasets for the prediction of several soil properties. Moreover, chemometrics- and spectroscopy-based soil calibrations need to be vigorously validated (Brown et al., 2005) to counteract the inherent soil spatial variability (Wang and Cao, 2013) and to ensure geographic model transferability (Reeves, 2010).

Shepherd and Walsh (2002) demonstrated successful diagnostic spectral screening tests for soil properties that were otherwise moderately calibrated to spectra. This suggested that spectral screening would allow the satisfactory classification of soils into basic quality classes. Kariuki et al. (2003) demonstrated strong correlations between infrared spectral parameters in the shortwave infrared (1400–2200 nm) and the established swelling potential indices of the Atterberg limits, CEC, and the coefficient of linear extensibility (COLE) tests. However, no quantification on the predictive performance for spectral screening tests has been reported for soil properties valuable for engineering land use.

The aim of this study is to assess the performance of MIR PLS analyses for the rapid characterization of selected key soil properties valuable for applications in engineering land use. Specifically, the goals of the study are (i) to develop internally validated MIR PLS models for the prediction of several key soil properties for mixed depth and separate depth datasets, and to validate the models using independent sample sets of similar soils; and (ii) to develop independently validated MIR spectra screening tests for various soil limitation classes applicable in civil works. A key question was whether the accuracy of the MIR PLS analyses is sufficient to allow the incorporation of the Atterberg limits and linear shrinkage data into the Africa Soil Information Service (AfSIS) baseline sample of sub-Saharan Africa (Shepherd, 2010; Vågen et al., 2013).

2. Materials and methods

2.1. Selection of sampling sites and field data collection

The study sites fall in an area within Lake Victoria basin (LVB) in the western part of Kenya covering approximately 46,400 km^2 and bound by latitudes 0°7′48″N and 0°24′36″S and longitudes 34°51′E and 35°43′12″E (Fig. 1). Two sets of soil samples were collected following a double-sampling approach (Shepherd and Walsh, 2007). A calibration sample set ($n=135$) was obtained from 46 sites established following a simplified version of the conditioned Latin hypercube sampling (Minasny and McBratney, 2006). At each site in the field, soil samples were collected at three depths (0–20, 20–50, and 50–100 cm) using a Dutch auger. The validation set ($n=120$) was obtained from a larger set ($n=417$) from two different and spatially separated sentinel sites (10×10 km^2 blocks) of lower Nyando (Lny) and Homa Bay (Hby) within LVB. Selection of the sentinel sites, sampling locations, and sample collection in the field followed the land degradation surveillance framework (LDSF) protocol (Vågen et al., 2013). Fig. 2 illustrates the layout and the distribution of the sampling plots in Lny. At each sampling plot in the field, composite samples were taken for three depths: 0–20, 20–50, and 50–100 cm. Prior to the analyses, bulk soil samples were air-dried at 40 °C for two weeks followed by gently crushing the samples and passing them through a 2-mm sieve. Subsamples were used for the analyses.

2.2. Sample preparation and spectral measurement

Spectral measurements for both calibration and validation sets were conducted using a High Throughput Screening device (HTS–XT) attached to a Tensor 27 spectrometer (Bruker Optics, Germany) customized for the MIR spectral range (4000–400 cm^{-1}) (Fig. 3). The measurement protocol elaborated by Terhoeven-Urselmans et al. (2010) was followed. About 5.0 g of each subsample of the air-dry <2 mm soil was ground to <0.5 mm using a natural stone pestle and mortar. The ground sample was thoroughly mixed and homogenized, and approximately 0.5 g was loaded into labeled wells in four replicates in aluminum micro-titer plates consisting of 96 wells (Fig. 4). The first two wells of the plate were used for the standard and blank, respectively, and the first sample was placed in the third well. Scanning was done sequentially for each well. The average reflectance of 32 scans per sample was transformed to absorbance and recorded using the Optics user's software (OPUS) (Bruker Optics, Germany).

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