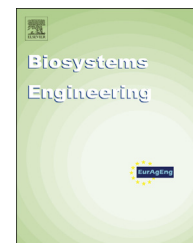




ELSEVIER

Available online at www.sciencedirect.com

ScienceDirect

journal homepage: www.elsevier.com/locate/issn/15375110

Research Paper

Rapid estimation of soil engineering properties using diffuse reflectance near infrared spectroscopy



Bernard K. Waruru^{a,*}, Keith D. Shepherd^b, George M. Ndegwa^c,
Peter T. Kamoni^a, Andrew M. Sila^b

^a Kenya Agricultural Research Institute (KARI), P.O. Box 1473, 00800 Nairobi, Kenya

^b World Agroforestry Centre (ICRAF), P.O. Box 30677, 00100 Nairobi, Kenya

^c Jomo Kenyatta University of Agriculture and Technology (JKUAT), P.O. Box 62000, 00200 Nairobi, Kenya

ARTICLE INFO

Article history:

Received 8 March 2013

Received in revised form

10 March 2014

Accepted 10 March 2014

Published online 29 March 2014

Keywords:

NIR spectroscopy

Soil engineering properties

Kenya

Materials testing involve complex reference methods and several soil tests have been used for indexing material functional attributes for civil engineering applications. However, conventional laboratory methods are expensive, slow and often imprecise. The potential of soil diffuse reflectance near infrared (NIR) spectroscopy for the rapid estimation of selected key engineering soil properties was investigated. Two samples sets representing different soils from across the Lake Victoria basin of Kenya were used for the study: A model calibration set ($n = 136$) was obtained using a conditioned Latin hypercube sampling, and a validation set ($n = 120$) using a spatially stratified random sampling strategy. Spectral measurements were obtained for air-dried (<2 mm) soil sub-samples using a Fourier-transform diffuse reflectance near infrared (NIR) spectrometer. Soil laboratory reference data were also obtained for liquid limit (LL), plastic limit (PL), plasticity index (PI), linear shrinkage (LS), coefficient of linear extensibility (COLE), volumetric shrinkage (VS), clay activity number (Ac), total clay content, air-dried moisture content, and cation exchange capacity (CEC). Soil reference data were calibrated to smoothed first derivative NIR spectra using partial least squares (PLS) regression. At the calibration stage, coefficient of determination for full cross-validation (R^2) of ≥ 0.70 was obtained for CEC, mc, LL, PI, LS, COLE and VS. Further independent validation gave $R^2 \geq 0.70$ and RPD (ratio of reference data SD and root mean square error of prediction) 1.7–2.2 for LL, PI, mc and CEC. The results suggested that NIR–PLS has potential for the rapid estimation of several key soil

Abbreviations and symbols: NIR, near infrared; LVB, Lake Victoria Basin; CEC, cation exchange capacity; tClay, total clay content; LL, liquid limit; PL, plastic limit; PI, plasticity index; LS, linear shrinkage; COLE, coefficient of linear extensibility; VS, volumetric shrinkage; Ac, clay activity number; mc, air-dried gravimetric moisture content; PLS, partial least squares regression; SWIR, shortwave infrared; UNEP, United Nations Environment Programme; HB, Homa Bay; LNY, lower Nyando; MPA, multi-purpose analyser; OPUS, optics users software; PCA, principal component analysis; BSI, British Standards Institution; Ca, calcium; Mg, magnesium; Na, sodium; K, potassium; SD, standard deviation; CV, coefficient of variability; looCV, leave-out-one cross-validation; H, Mahalanobis distance; PC, principal component; R^2 , coefficient of determination; RMSECV, root mean square error of cross-validation; RMSEP, root mean square error of prediction; RPD, ratio of standard deviation of reference values to RMSECV/RMSEP; SOC, soil organic carbon.

* Corresponding author. Tel.: +254 02 0729 556169.

E-mail addresses: bkwaruru@gmail.com, bkwaruru@yahoo.com (B.K. Waruru).

<http://dx.doi.org/10.1016/j.biosystemseng.2014.03.003>

1537-5110/© 2014 The Authors. Published by Elsevier Ltd. on behalf of IAgrE. This is an open access article under the CC BY-NC-ND license (<http://creativecommons.org/licenses/by-nc-nd/3.0/>).

engineering properties. Further work should focus on extending calibration libraries using more diverse soil types and testing alternative infrared diffuse reflectance based methods.

© 2014 The Authors. Published by Elsevier Ltd. on behalf of IAgre. This is an open access article under the CC BY-NC-ND license (<http://creativecommons.org/licenses/by-nc-nd/3.0/>).

1. Introduction

Different materials testing systems (e.g., double-oedometer, triaxial, shear, Proctor, and California Bearing Ratio test) have been devised in an effort to produce empirical standard evidence to evaluate the ability of any given soil to perform. However, these tests use complicated equipment, require highly skilled operators and use also large amounts of sample materials. Several soil tests have been used to provide rapid indicators of soil functional capacity for preliminary geotechnical site investigations. These include soil particle size analysis, Atterberg or consistency limits, linear shrinkage and derived parameters. Others include gravimetric moisture content (mc) and cation exchange capacity (CEC) (Bell, 2000, chap. 3; Bell, Culshaw, & Northmore, 2003; Bowles, 1992, chap. 1; Fratta, Aquettant, & Roussel-Smith, 2007, chap. 3). However, conventional laboratory methods are expensive, slow and often imprecise. The methods also involve single soil tests and physical destruction of the soil system and require the use of (environmentally harmful) chemical extractants. Analysis of soil using diffuse reflectance infrared technology is an alternative well known for its rapidity, simplicity, precision and cost-effectiveness (Viscarra Rossel & McBratney, 1998).

Diffuse reflectance near infrared spectroscopy (NIR) has extensively been applied for analyses in very diverse fields including, agriculture, geology, medicine, and soil science (Shepherd & Walsh, 2007). In soil science, numerous studies (Stenberg, Viscarra-Rossel, Mouazen, & Wetterlind, 2010) have demonstrated that the NIR spectral range combined with a multivariate calibration method could be used as a non-destructive rapid analytical technique to simultaneously estimate several soil compositional constituents and soil quality attributes with acceptable accuracy in a very short time. NIR is additionally adaptable for field-based and 'on-the-go field use (Stenberg et al., 2010) important for rapid preliminary geotechnical investigations.

There already is some evidence of the utility of NIR in predicting soil engineering properties (Kariuki, Van der Meer, & Siderius, 2003; Kariuki, Van der Meer, & Verhoef, 2003). However, no studies were available on the potential of NIR combined with partial least squares (PLS) regression for rapid estimation of consistency (Atterberg) limits (liquid limit-LL, plastic limit-PL, plasticity index-PI), linear shrinkage (LS), and associated coefficient of linear extensibility (COLE) and volumetric shrinkage (VS). Kariuki, Van der Meer, and Siderius (2003) achieved satisfactory classification of soils into broad swell-potential categories by relating spectral absorption features (feature position, asymmetry and depth) in the shortwave infrared (SWIR: 1300–2500 nm) with threshold values of shrink-swell indicator soil properties (including CEC, PI and COLE tests). Kariuki, Van der Meer, and Verhoef (2003)

found strong relationship of SWIR spectral absorption features (asymmetry at 1400 and 2200 nm) with soil activity (ratio CEC:% clay), a commonly applied engineering index for estimation of CEC, suggesting great potential of NIR for estimation of CEC. However, geographic transferability continues to challenge the widespread use and application of soil infrared spectroscopic studies (Reeves, 2010; Stenberg et al., 2010), a result of application of less rigorous validation strategies (Brown, Bricklemeyer, & Miller, 2005).

Few studies using NIR-PLS for soil analyses have used completely independent validation data although Dardenne, Sinnaeve, and Baeten (2000) found that the veracity of chemometric-based models could only be established using totally independent test sets, preferably collected after model calibration. Nanni and Demattè (2006) interchanged surface and subsurface samples data sets as calibration and validation sets for estimation of several soil properties, a strategy that Brown et al. (2005) refer to as pseudo-independent validation. Sorensen and Dalsgaard (2005) used samples sets from the relatively more homogeneous surface horizon and a restricted clay range (<26%) for independent prediction of total clay content. Independent validation data sets drawn from the target population are required to establish true predictive ability and robustness of a spectral test and evaluate its fitness for purpose. This study therefore aimed to assess performance of NIR-PLS for rapid estimation of several soil properties that are commonly used in materials stability indices for civil engineering applications using an independent validation data set drawn from the same geographical area. A key question was whether the prediction accuracy would be sufficient for engineering applications and standards.

2. Materials and methods

2.1. Sample collection

Two sets of soil samples were collected from an area that fall within Lake Victoria Basin (LVB) in the western part of the Republic of Kenya covering approximately 46,400 km² and bound by latitudes 0°7'48"N, 0°24'36"S and longitudes 34°51'E, 35°43'12"E and the 1400 m above sea level contour (Fig. 1). Sampling sites for the calibration set ($n = 136$) (Fig. 1) were established following a simplified version of the conditioned Latin hypercube sampling (Minasny & McBratney, 2006).

The validation set ($n = 120$) was obtained from two different and spatially separated sentinel sites (10 × 10 km blocks, see UNEP (2012) for sentinel site sampling design), Homa Bay (HB) and Lower Nyando (LNY) within LVB of Kenya (see inset in Fig. 1), one year after collection of the calibration set. Selection of the sites and sample collection in the field

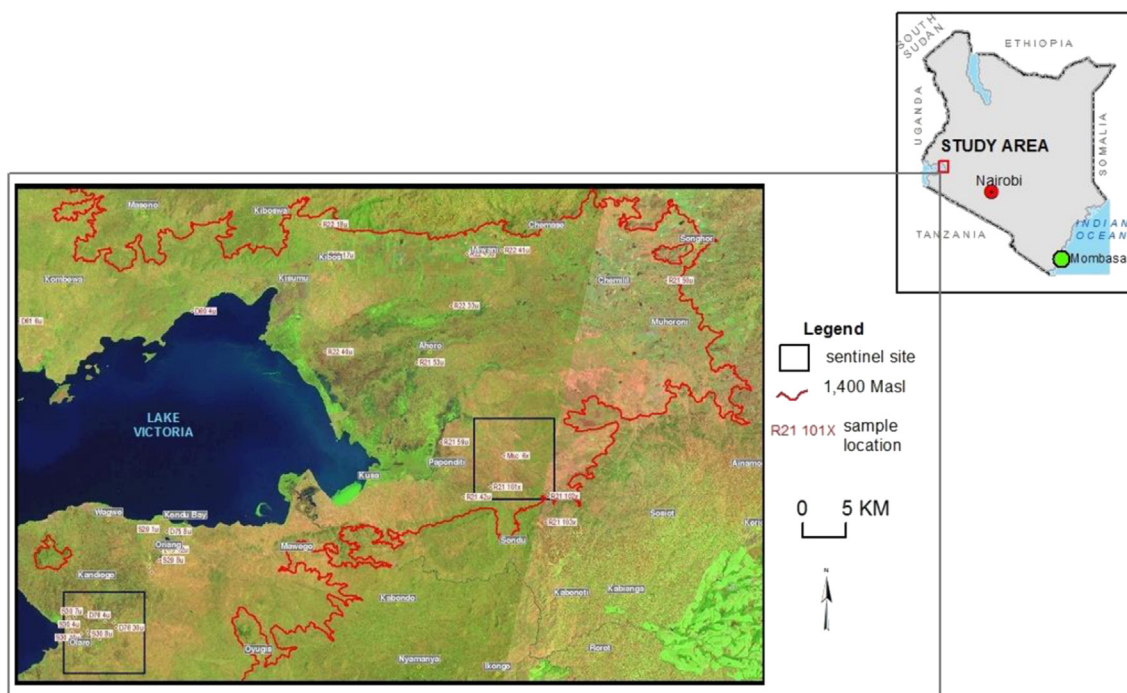


Fig. 1 – Distribution of calibration set sampling sites within Lake Victoria basin of Kenya (also indicating two sentinel sites used for obtaining validation samples).

followed the land degradation surveillance framework protocol (Walsh & Vagen, 2006). Figure 2 illustrates layout and distribution of sampling plots in HB. In the field, samples were collected at three depths (0.0–0.2, 0.2–0.5, 0.5–1.0 m) using a Dutch soil auger. Prior to analyses, bulk soil samples were air-dried at 40 °C for two weeks followed by gently crushing to pass a 2-mm sieve. Sub-samples were then used for various analyses.

2.2. Spectral measurements

Spectral measurements were conducted using a Fourier-transform diffuse reflectance spectrometer (Multi-Purpose Analyser (MPA), Bruker Optics, Germany) customised for NIR (12,500–3600 cm^{-1}). Measurements were made using resolution of 8 cm^{-1} , taking an average of 32 scans. Reflectance spectra were transformed to absorbance spectra and recorded using the Optics Users Software (OPUS) (Bruker Optics, Germany) (Shepherd, 2010). Principal component analysis (PCA) of the NIR spectra for the combined HB and LNY sample set ($n = 417$) was used to select representative 120 (~25%) samples that were used to provide reference data and spectral variables for validation.

2.3. Development of soil reference data

Soil physical and chemical analyses were conducted using standard laboratory methods as reported by Shepherd and Walsh (2002) for development of calibration and validation models. Air-dried soil moisture content (mc %) was determined by gravimetric method. Total clay content (tClay, %)

was determined by hydrometer method following Gee and Bauder (1986). Soil effective CEC ($\text{cmol}(+) \text{kg}^{-1}$) was obtained as the numeric sum of exchangeable Ca, Mg, Na, and K. Determination of Atterberg limits LL and PL (%) and LS (%) followed British Standards Institution (BSI: 1377, 1975, chap. 2). The PI (%) was computed as the numerical difference between LL and PL. Clay activity number (Ac, unit) was obtained as the ratio of PI to tClay. The COLE (unit) and VS (%) were computed using LS data as reported by Igwe (2003).

Repeatability and intra-laboratory reproducibility tests were conducted for Atterberg limits and linear shrinkage. Repeatability was assessed using the coefficient of variability (CV), expressed in percentage [$\% \text{CV} = (\text{SD}/\text{mean}) \times 100$], where SD is the standard deviation of the measurements.

2.4. Soil properties

Soil depths (0.0–0.2, 0.2–0.5, 0.5–1.0 m) were combined for both calibration and validation samples sets. This presented a wide range in soil property data (Table 1). For both samples sets PL and Ac were highly skewed and natural log (ln) transformation was applied to reduce skewness. Soil mc and CEC had slight skewness and square-root transformation was applied. Raw data distribution for tClay, LL, PI, LS, COLE and VS approximated a Gaussian distribution and so no transformation was used.

Soil property data for the validation sample set were within the range of the calibration set for all soil properties except for CEC and tClay (Table 1), satisfying a key requirement for a successful NIR PLS analysis (Stenberg et al., 2010). Data range for CEC was 3.41–67.0 and 4.79–76.4 ($\text{cmol}(+) \text{kg}^{-1}$) in the

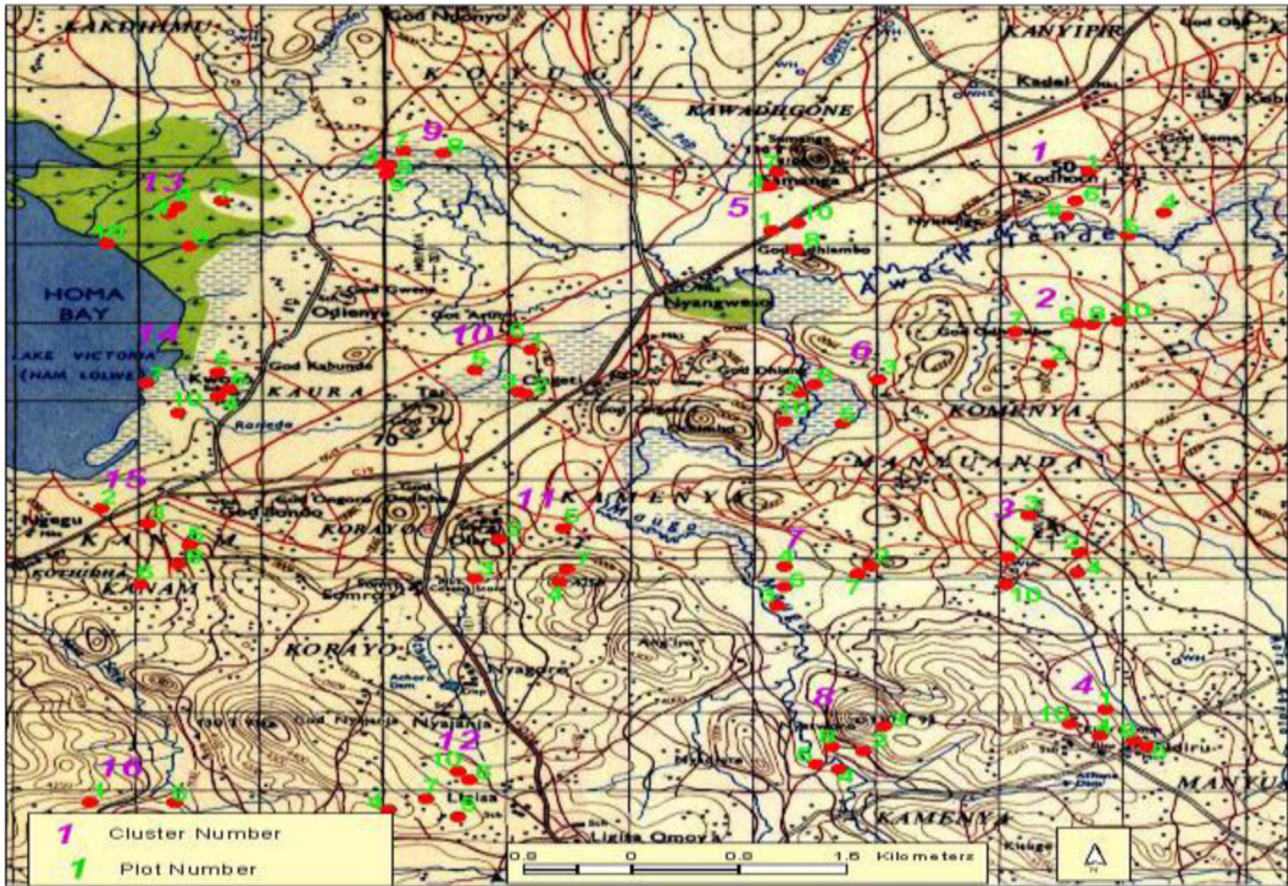


Fig. 2 – Sentinel site indicating distribution of clusters and sampling plots for validation set (plots marked in red were priority locations).

calibration and validation sets respectively. The tClay indicated 9.0–61.0 and 7.0–81.0 (%) in the calibration and validation sets respectively.

PCA scores plot of NIR spectra for combined calibration ($n = 136$) and validation ($n = 417$) samples (Fig. 3) indicated that the majority of validation samples (marked with 'v') were within range of calibration samples (marked with 'c'), affirming that the two sets belonged to the same soil population.

2.5. Calibration of soil properties

Prior to calibration development, transformed reference data were mean-centred and then scaled ($1/SD$). Absorbance spectra were transformed using Savitzky–Golay first derivative and a smoothing factor. Only the <8000 and $>4000\text{ cm}^{-1}$ spectral bands were used, to exclude excessive noise at the shorter wavelengths. Spectral outliers were checked in the

Table 1 – Soil engineering properties for calibration samples set (corresponding data for validation set is shown in parenthesis).

| Property | min | 25% | 50% | 75% | max | SD |
|----------|-------------|-------------|-------------|-------------|--------------|-------------|
| CEC | 3.41 (4.8) | 16.6 (16.8) | 26.4 (30.3) | 36.6 (43.8) | 67.0 (76.4) | 14.0 (16.6) |
| tClay | 9.0 (7.0) | 25.0 (30.5) | 41.0 (47.0) | 51.0 (61.0) | 61.0 (81.0) | 14.8 (17.0) |
| LL | 21.8 (22.2) | 40.5 (44.3) | 52.7 (60.8) | 66.0 (73.5) | 90.7 (96.7) | 18.7 (18.5) |
| PL | 10.8 (11.4) | 17.1 (18.1) | 21.2 (22.4) | 25.8 (26.9) | 39.3 (45.3) | 6.9 (6.3) |
| PI | 5.5 (8.4) | 21.8 (22.8) | 30.1 (35.6) | 41.1 (47.0) | 62.8 (66.1) | 14.0 (14.5) |
| LS | 2.9 (3.6) | 9.9 (11.4) | 12.1 (14.3) | 14.3 (15.7) | 21.2 (20.0) | 4.2 (3.8) |
| COLE | 0.03 (0.04) | 0.11 (0.13) | 0.14 (0.17) | 0.17 (0.19) | 0.27 (0.25) | 0.05 (0.05) |
| VS | 9.1 (11.5) | 36.6 (43.9) | 47.5 (58.9) | 58.8 (67.3) | 104.3 (95.3) | 20.6 (19.4) |
| Ac | 0.4 (0.3) | 0.7 (0.6) | 0.8 (0.7) | 1.0 (0.9) | 1.8 (1.9) | 0.3 (0.3) |
| mc | 0.7 (0.7) | 4.4 (4.2) | 6.5 (6.9) | 8.3 (9.1) | 15.9 (13.4) | 3.1 (3.3) |

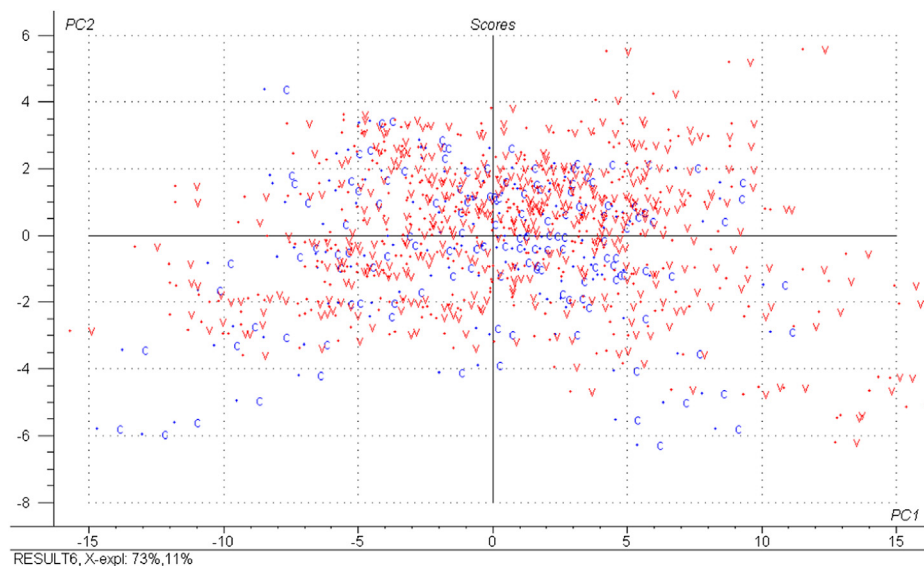


Fig. 3 – PCA scores plot (PC1 vs PC2) for NIR spectra of combined calibration and validation samples sets (PCA was run in Unscrambler using leverage correction for model validation. Validation samples (v) outside the span of calibration samples (c) were visually identified as potential spectral outliers).

calibration set using Mahalanobis distance (Næs, Isaksson, Fern, & Davies, 2002; Shepherd & Walsh, 2002). PCA scores plot of derivative NIR spectra was used to compute Robust Mahalanobis distances (H). Samples with $H > 12$ were considered potential outliers. The transformed spectra were calibrated to each of the soil property reference values by PLS 1. Calibrations were made with leave-out-one cross-validation (looCV). The Unscrambler software version 9.2 (Camo Software ASA, Oslo, Norway) was used for data pretreatment and calibration. The calibration procedure in The Unscrambler is well described by other workers (Canasveras, Barron, Del Campillo, Torrent, & Gomez, 2010; Stenberg, 2010). In The Unscrambler, full model with a maximum 20 principal components (PCs) was set; however, the optimal number of PCs to be used for each property was determined using residual variances (CAMO ASA, 1998). The predictive ability of the models was evaluated by the coefficient of determination (R^2), the root mean square error of cross-validation (RMSECV) and the RPD (ratio of SD of calibration reference values to RMSECV). The RMSECV was calculated as follows:

$$\text{RMSECV/RMSEP} = \left[\sum (y - x)^2 / n - 1 \right]^{1/2} \quad (1)$$

where y is the predicted value by NIR-PLS technique, x is the reference value, and n is the total number of samples.

The looCV calibration models with $R^2 > 0.3$ (Saeys, Xing, De Baerdemaeker, & Ramon, 2005) were further validated using similarly preprocessed reference and NIR spectral data for the samples from independent sites. The predictive ability of the models was evaluated by the coefficient of determination (R^2), the root mean square error of prediction (RMSEP), and the RPD (ratio of SD of validation reference values to RMSEP). RMSEP was calculated using Eq. (1). The Unscrambler software was used for the predictions. Calibration and independent

validation statistics (coefficient of determination, RMSECV, RMSEP, and RPD) were given for back-transformed data.

A reference value outlier sample was defined as sample whose difference between predicted and measured value for a soil property was greater than $3 \times \text{RMSECV/RMSEP}$ (Islam, Singh, & McBratney, 2003). For the validation set, the number of samples detected as reference value outliers were 1 for tClay, 2 for mc, LL, PI, PL, LS and VS, 3 for CEC and COLE. Samples with spurious (negative) predictions (1 for LL, PI and COLE; 2 for LS and VS) were also considered outliers. The outlier samples were excluded from predictions and the model statistics (R^2 and RMSEP) recalculated. Prediction for PL was optimised with removal also of non-plastic soils (total six samples). One sample had inadequate soil material for mechanical tests (LL, PI, PL, LS COLE and VS). Five spectral outliers were removed (Robust Mahalanobis > 12) in the calibration set but had no impact on looCV models.

Table 2 – Repeatability (% CV) for soil Atterberg limits and linear shrinkage.

| Test | B ^a | C ^a | L ^a | N ^a | W ^a | J ^a | E ^a |
|------|----------------|----------------|----------------|----------------|----------------|----------------|----------------|
| LL% | 2.8 | 4.4 | 3 | 1.7 | 14.3 | 1.5 | 4.4 |
| PL% | 20.5 | 15.3 | 16.2 | 6.2 | 22.6 | 3.3 | 9.2 |
| PI% | 23.8 | 21.3 | 22.3 | 9.9 | 4.7 | 3.9 | 6.2 |
| LS% | 3.4 | 15.8 | 4.1 | 5.3 | 4.6 | 2.9 | 14.8 |
| COLE | 3.9 | 17.7 | 4.7 | 5.9 | 4.6 | 3.3 | 16.4 |
| VS% | 1.4 | 5.9 | 1.7 | 1.8 | 1 | 1.3 | 5.2 |

^a Codes: B, C, E, J, L, N, and W identify the seven different operators used for the test; column data indicate repeatability for individual operators, whereas row data indicate reproducibility across the seven different operators; the test sample was a friable red clay loam soil of moderate plasticity (LL = 45.3, PL = 29.8, PI = 15.5).

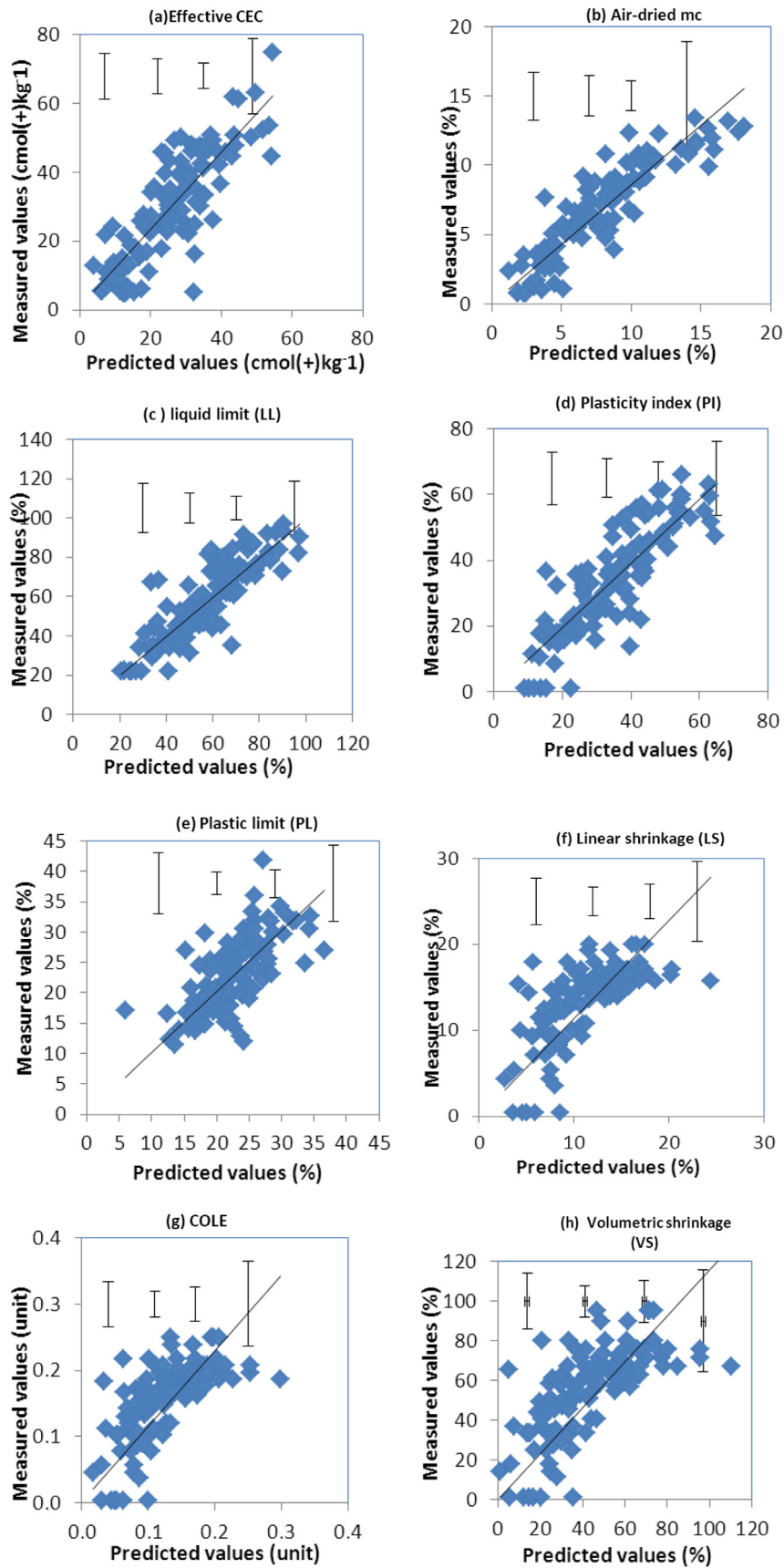


Fig. 4 – Scatterplot comparison of measured and predicted values for different soil engineering properties. Cross-validated models developed with partial least squares (PLS) regression were further tested using an independent sample set. Validation statistics were as follows: (a) CEC ($R^2 = 0.70$, RMSEP = 9.6, $n = 117$); (b) mc ($R^2 = 0.80$, RMSEP = 1.95, $n = 118$); (c)

3. Results and discussion

3.1. Soil properties

Atterberg limits and linear shrinkage gave variable measurement precision (Table 2). LL indicated % CV 1.5–14.3, but was better reproduced by six of the seven operators (% CV 1.5–4.4). PL, PI and LS presented greater variability across the different operators.

The quality of reference data is among the key factors influencing performance of NIR models (Stenberg et al., 2010). Low repeatability and reproducibility for both Atterberg limits and LS (Table 2), suggested potential for low quality reference data. Several workers (Genot et al., 2011; Shepherd, Vanlauwe, Gachengo, & Palm, 2005) have shown that NIR measurements are more repeatable than wet chemistry.

3.2. Calibration of soil properties

NIR PLS statistics for CEC was excellent (R^2 0.80, RMSECV 5.9 cmol (+) kg⁻¹, RPD 2.4) and better than R^2 0.75 reported by Islam et al. (2003) using samples from different soil horizons. This was attributed to strong association of CEC with spectrally active soil clay mineralogy (reflected by tClay), soil organic carbon (SOC) and mc (Chang, Laird, Mausbach, & Hurburgh, 2001; Stenberg et al., 2010). Estimation of tClay was weak (R^2 0.5, RMSECV 11.0%, RPD 1.4) although texture exhibits a primary response to NIR spectra (Chang et al., 2001). This was attributed to the negative effect on the path of light and reflectance spectra occasioned by variation in soil particle size, shape, and arrangement for different samples in the calibration set (Stenberg et al., 2010). Estimation of LL (R^2 0.83, RMSECV 6.9%, RPD 2.7) and mc (R^2 0.83, RMSECV 1.3%, RPD 2.4) was excellent, attributed to high resonance of soil water with characteristic intense water absorption bands in the NIR region (Mouazen, De Baerdemaeker, & Ramon, 2006). Prediction of mc was similar to R^2 0.85 and RPD 2.0 attained by Islam et al. Weak (R^2 0.5, RMSECV 4.1%) but reliable (RPD 1.7) performance was observed for PL. Performance for PI (R^2 0.7, RMSECV 7.6%, RPD 1.9) was between that for LL and PL (PI = LL - PL). Estimation of LS and associated COLE and VS was reliable (R^2 0.7, RPD 1.8–2.0) and prediction errors 2.1%, 0.03, and 10.9%, respectively. This was attributed to association of LS with soil mc allowing secondary response (indirect co-variation) of LS with NIR spectra (Stenberg et al.). NIR PLS could, however, not predict Ac ($R^2 < 0.3$, RPD 1.3). Poor prediction of PL and Ac was attributed to poor quality reference data (Table 2) (Stenberg et al.). Determination of Ac was, for example, susceptible to error propagation in the determination of tClay, LL, and PL.

The NIR PLS looCV analyses suggested great potential for rapid estimation of soil engineering properties CEC, mc, LL, PL, PI, LS, COLE and VS (R^2 0.53–0.83; RPD 1.8–2.7). However, looCV could overestimate the predictive performance of a

model (Brown et al., 2005). Therefore, the looCV models were further tested using spectra of an independent sample set.

3.3. Independent validation

Good independent validation (R^2 0.7, RMSEP 9.6 cmol (+) kg⁻¹, RPD 1.7) was observed for the CEC model (Fig. 4(a)). This reflected the robust relationship between CEC and spectrally active soil components (mc, SOC, and clay mineralogy) (Stenberg et al., 2010). These results were comparable with R^2 0.64 and RPD 1.6 reported by Islam et al. (2003) using the less rigorous separate test set validation strategy. Nanni and Demattè (2006) reported R^2 0.4–0.7 for CEC by interchanging surface and subsurface samples sets data sets as calibration and validation sets respectively.

The observed robust (R^2 0.80, RMSEP 2.0%, RPD 2.0) model for mc (Fig. 4(b)) was attributed to characteristic strong moisture absorption features in the NIR spectral region (Mouazen et al., 2006). Prediction of tClay was weak and unreliable (R^2 0.5, RMSEP 16.0%, RPD 1.1). Variation in soil particle size, shape, and arrangement in different soils samples results in highly variable NIR PLS performance for individual textural parameters (Bellon-Maurel & McBratney, 2011; Stenberg et al., 2010). The results compared well with R^2 0.5–0.8 reported for tClay by Nanni and Demattè (2006).

NIR PLS models for LL (R^2 0.74, RMSEP 9.9%, RPD 2.2) and PI (R^2 0.73, RMSEP 8.3%, RPD 1.9) (Fig. 4(c) and (d)) were robust, reflecting stability of the models for reference values at standardised moisture content. We presume that aquaphotomics (water–light interactions) recently introduced by Stenberg (2010) in soil spectroscopic studies could be responsible for calibration and stability of the otherwise spectrally non-responsive mechanical properties. The models for PL, LS, COLE and VS (Fig. 4(e), (f), (g), and (h) respectively) were however less robust (R^2 0.46, RPD 1.1–1.7), probably due to low quality reference values associated with reference methods (Table 2).

The model results showed that NIR could provide robust models for rapid estimation of LL, PI, mc and CEC (R^2 0.7–0.8; RPD 1.7–2.2). The technique presented modest but reliable independent estimation of PL, LS, COLE and VS (R^2 0.5, RPD 1.1–1.7). Canasveras et al. (2010) found that for modest RPD values between 1.5 and 1.69 for prediction of soil stability attributes, the spectral based predictors are useful for screening purposes such as discriminating between low, medium and high stability classes. Comparing calibration and independent validation statistics, the observed general lowering of model performance is to be expected given inherent challenges with geographic transferability of spectra-based chemometrics models (Reeves, 2010; Stenberg et al., 2010).

3.3.1. NIR prediction error and fitness for purpose

Table 3 present a statistical description (mean, SD and data range) of the observed soil property data analysed using

LL ($R^2 = 0.74$, RMSEP = 9.9, $n = 116$); (d) PI ($R^2 = 0.73$, RMSEP = 8.3, $n = 116$); (e) PL ($R^2 = 0.45$, RMSEP = 4.6, $n = 111$); (f) LS ($R^2 = 0.46$, RMSEP = 4.13, $n = 115$); (g) COLE ($R^2 = 0.46$, RMSEP = 0.05, $n = 115$); (h) VS ($R^2 = 0.47$, RMSEP = 20.8, $n = 115$). The target (1:1) regression line is shown for each plot. Error bars illustrate the standard error (SE) of the predicted values for the different quartiles.

Table 3 – Statistical description of the observed soil property data analysed using conventional methods of analyses and their near infrared (NIR) partial least squares (PLS) independent predictions.

| Property | Observed | | Predicted | |
|----------|-----------------|------------|-----------------|------------|
| | Mean \pm SD | Data range | Mean \pm SD | Data range |
| CEC | 30.6 \pm 16.6 | 4.8–76.4 | 26.1 \pm 11.7 | 4.2–54.7 |
| mc | 6.8 \pm 3.3 | 0.7–13.4 | 7.92 \pm 3.8 | 1.25–18.1 |
| tClay | 44.7 \pm 17.0 | 7.0–81.0 | 34.0 \pm 11.2 | 1.4–52.4 |
| LL | 56.0 \pm 22.2 | 22.2–96.7 | 57.7 \pm 17.6 | 20.5–97.2 |
| PI | 33.7 \pm 16.1 | 8.4–66.1 | 35.1 \pm 13.5 | 9.01–64.6 |
| PL | 21.8 \pm 8.0 | 11.4–45.3 | 22.3 \pm 5.1 | 6.0–36.7 |
| LS | 12.9 \pm 4.7 | 3.6–20.0 | 11.0 \pm 4.2 | 2.7–24.4 |
| COLE | 0.2 \pm 0.06 | 0.037–0.3 | 0.13 \pm 0.05 | 0.02–0.3 |
| VS | 53.8 \pm 22.7 | 11.5–95.3 | 43.3 \pm 21.4 | 1.2–110.5 |

conventional methods and their independently validated NIR PLS predictions. In most cases there was a good correspondence between predicted and observed ranges. However, some of the estimates were less accurate than those obtained by routine laboratory methods.

Given the relative speed and cost of this approach and the large local variation of soil properties, we suggest that the ability to analyse large number of samples involving multiple variables at finer sampling intervals using the spectroscopic technique may in some circumstances outweigh the loss in analytical precision. NIR is also known to be more reproducible (precise) than the reference methods and this could convey a distinct advantage. However the key criterion for judging acceptable prediction accuracy is fitness for purpose.

A classification of soils into broad limitation classes is often adequate, for example, for rapid geotechnical site investigations, especially for earthen works and foundations for small buildings construction (Bell, 2000, chap. 3; Hazelton & Murphy, 2007, chap. 3; McKenzie, Coughlan, & Cresswell, 2002). However, the required accuracy for a civil construction project, for example, may be much more stringent than, say, rating based on limitation thresholds. For applications where high accuracy is required, NIR may not always meet the requirements but can still be proposed as a triage system to help with sampling and analytical decisions. If NIR predicted values are in a low or high range relative to a decision cut-off value (e.g. an acceptable linear shrinkage value) then the evidence may be sufficient to make a decision (e.g. there is a high probability that the site is stable or unstable). However if predictive values lie within a range that straddles the cut-off limit, then samples may be recommended for conventional analysis. A two-phase sampling approach can also be adopted, whereby a subset of samples is selected to verify that the NIR predictions are sufficiently accurate for the specific application. The use of NIR in creating diagnostic screening tests was illustrated by Shepherd and Walsh (2002).

4. Conclusions

The application of NIR together with PLS was tested for rapid estimation of key soil engineering properties CEC, mc, tClay, LL, PI, PL, LS, COLE, VS and Ac. From the results obtained, NIR-PLS demonstrated robust models (R^2 0.7, RPD \geq 1.7) for rapid estimation of LL, PI, mc and CEC for an independent

sample set. NIR PLS models for PL, LS, COLE and VS (R^2 0.5, RPD 1.1–1.7) provided quality indices valuable for sorting soils into stability classes for preliminary geotechnical investigations. However, for the studied soils the technique showed minimal potential for the characterisation of Ac. It can be concluded that NIR combined with PLS has great potential for rapid estimation of LL, PI, mc and CEC. This affirms the potential role that reflectance spectroscopy could play to provide simple, rapid, and cost-effective tests for soil engineering properties, especially where large sample sizes and multiple variables are to be analysed. Further work should focus on extending calibration libraries using more diverse soil types and also alternative infrared diffuse reflectance based methods. Direct calibration of “fitness for use” classes for engineering applications to spectral data should also be considered.

Acknowledgement

The authors thank Ms Akinyi at Materials Testing and Research laboratory for support with mechanical analyses. We thank Elvis Weullow and the team at the Soil and Plant Spectral Diagnostic laboratory at ICRAF for soil processing and spectral measurements. This study was jointly funded by Kenya Agricultural Research Institute (KARI), Kenya Agricultural Productivity Project (KAPP) and the World Agroforestry Centre (ICRAF) Science Domain 4 – Land Health.

REFERENCES

- Bell, F. G. (2000). *Engineering properties of soils and rocks* (4th ed.). Osney Mead, Oxford: Blackwell Science Ltd.
- Bell, F. G., Culshaw, M. G., & Northmore, K. J. (2003). The metastability of some gull-fill materials from Allington, Kent, UK. *Quarterly Journal of Engineering Geology and Hydrogeology*, 36, 217–229.
- Bellon-Maurel, V., & McBratney, B. (2011). Review: near-infrared (NIR) and mid-infrared (MIR) spectroscopic techniques for assessing the amount of carbon stock in soils – critical review and research perspectives. *Soil Biology and Biochemistry*, 43, 1398–1410.
- Bowles, J. E. (1992). *Engineering properties of soils and their measurement* (4th ed.). New York: McGraw-Hill.

- British Standard Institute (BSI). (1975). *Methods of test for soils for civil engineering purposes*. Park Street, London: British Standard Institution.
- Brown, D. J., Bricklemeyer, R. S., & Miller, P. R. (2005). Validation requirements for diffuse reflectance soil characterization models with a case study of VNIR soil C prediction in Montana. *Geoderma*, 129, 251–267.
- CAMO ASA Inc. (1998). *The unscrambler user manual*. Corvallis, OR: CAMO Inc.
- Canasveras, J. C., Barron, V., Del Campillo, M. C., Torrent, J., & Gomez, J. A. (2010). Estimation of aggregate stability indices in Mediterranean soils by diffuse reflectance spectroscopy. *Geoderma*, 158, 78–84.
- Chang, C. W., Laird, D. A., Mausbach, M. J., & Hurburgh, C. R. (2001). Near-infrared reflectance spectroscopy–principal components regression of soil properties. *Soil Science Society of America Journal*, 65, 480–490.
- Dardenne, P., Sinnaeve, G., & Baeten, V. (2000). Multivariate calibration and chemometrics for near infrared spectroscopy: which method? *Journal of Near Infrared Spectroscopy*, 8(4), 229–237.
- Fratta, D., Aquettant, J., & Roussel-Smith, L. (2007). *Introduction to soil mechanics laboratory testing*. Boca Raton: CRC Press.
- Gee, G. W., & Bauder, J. W. (1986). Particle-size analysis. In A. Klute (Ed.) (2nd ed.) *Methods of soil analysis Part 1. Physical and mineralogical methods* (pp. 383–411). Madison, WI: American Society of Agronomy.
- Genot, V., Colinet, G., Bock, L., Vanvyve, D., Reusen, Y., & Dardenne, P. (2011). Near infrared reflectance spectroscopy for estimating soil characteristics valuable in the diagnosis of soil fertility. *Journal of Near Infrared Spectroscopy*, 19, 117–138.
- Hazelton, P., & Murphy, B. (2007). *Interpreting soil test results: What do all the numbers mean?* (pp. 31–47).
- Igwe, C. A. (2003). Shrink – swell potential of floodplain soils in Nigeria in relation to moisture content and mineralogy. *Journal of International Agrophysics*, 17, 47–55.
- Islam, K., Singh, B., & McBratney, A. B. (2003). Simultaneous estimation of several soil properties by ultra-violet, visible, and near-infrared spectroscopy. *Australian Journal of Soil Research*, 41, 1101–1114.
- Kariuki, P. C., Van der Meer, F. D., & Siderius, W. (2003). Classification of soils based on engineering indices and spectral data. *International Journal of Remote Sensing*, 24(12), 2567–2574.
- Kariuki, P. C., Van der Meer, F. D., & Verhoef, P. N. W. (2003). Cation exchange capacity (CEC) determination from spectroscopy. *International Journal of Remote Sensing*, 24(1), 161–167.
- McKenzie, N., Coughlan, K., & Cresswell, H. (2002). *Soil shrinkage* (Chapters 18 and 19). In *Soil physical measurement and interpretation for land evaluation* (pp. 240–270). CSIRO Publishing.
- Minasny, B., & McBratney, A. B. (2006). A conditioned Latin hypercube method for sampling in the presence of ancillary information. *Computers & Geosciences*, 32, 1378–1388.
- Mouazen, A. M., De Baerdemaeker, J., & Ramon, H. (2006). Characterization of soil water content using measured visible and near infrared spectra. *Soil Science Society of America Journal*, 70, 1295–1302.
- Naes, T., Isaksson, T., Fern, T., & Davies, T. (2002). *A user-friendly guide to multivariate calibration and classification*. Chichester, UK: NIR Publications.
- Nanni, M. F., & Demattè, J. A. (2006). Spectral reflectance methodology in comparison to traditional soil analysis. *Soil Science Society of America Journal*, 70, 393–407.
- Reeves, J. B. (2010). Review: near- versus mid-infrared diffuse reflectance spectroscopy for soil analysis emphasizing carbon and laboratory versus on-site analysis: where are we and what needs to be done? *Geoderma*, 158, 3–15.
- Saeys, W., Xing, J., De Baerdemaeker, J., & Ramon, H. (2005). Comparison of transmittance and reflectance to analyse hog manures. *Journal of Near Infrared Spectroscopy*, 13, 99–108.
- Shepherd, K. D. (2010). Soil spectral diagnostics – infrared, X-ray and laser diffraction spectroscopy for rapid soil characterization in the Africa Soil Information Service. In *19th World congress of soil science, soil solutions for a changing world*, 1–6 August 2010, Brisbane, Australia. Published on CDROM.
- Shepherd, K. D., Vanlauwe, B., Gachengo, C. N., & Palm, C. A. (2005). Decomposition and mineralization of organic residues predicted using near infrared spectroscopy. *Plant and Soil*, 277, 315–333.
- Shepherd, K. D., & Walsh, M. G. (2002). Development of reflectance spectral libraries for characterization of soil properties. *Soil Science Society of America Journal*, 66, 988–998.
- Shepherd, K. D., & Walsh, M. G. (2007). Infrared spectroscopy – enabling an evidence-based diagnostic surveillance approach to agricultural and environmental management in developing countries. *Review Journal of Near Infrared Spectroscopy*, 15, 1–19.
- Sorensen, L. K., & Dalsgaard, S. (2005). Determination of clay and other soil properties by near infrared spectroscopy. *Soil Science Society of America Journal*, 69, 159–167.
- Stenberg, B. (2010). Effects of soil sample pretreatments and standardized rewetting as interacted with sand classes on Vis-NIR predictions of clay and soil organic carbon. *Geoderma*, 158, 15–22.
- Stenberg, B., Viscarra-Rossel, R. A., Mouazen, A. M., & Wetterlind, J. (2010). Visible and near-infrared spectroscopy in soil science. *Review Advances in Agronomy*, 107, 163–215.
- UNEP. (2012). *Land health surveillance: An evidence-based approach to land ecosystem management. Illustrated with a case study in the West Africa Sahel*. Nairobi: United Nations Environment Programme.
- Viscarra Rossel, R. A., & McBratney, A. B. (1998). Soil chemical analytical accuracy and costs: implications from precision agriculture. *Australian Journal of Experimental Agriculture*, 38, 765–775.
- Walsh, M. G., & Vagen, T.-G. (2006). *Land degradation surveillance framework (LDSF) – Guide to field sampling and measurement procedures*. Nairobi, Kenya: World Agroforestry Centre (ICRAF).