Vis-NIR Spectroscopy for Determining Physical and Chemical Soil Properties: An Application to an Area of Southern Italy

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Abstract: The development of rapid, accurate, cost effective methods to determine soil physical and chemical properties is important for sustainable land management. In the last two to three decades, the interest in using visible and near infrared (Vis-NIR) spectroscopy as an alternative method for determining soil properties has increased. To obtain reliable predictions of soil properties, multivariate calibration techniques such as Partial Least Squares Regression (PLSR) are commonly used to correlate the spectra with the chemical, physical and mineralogical properties of soils.

The objective of the paper was to assess the potential of Vis-NIR spectroscopy coupled with PLSR to determine soil chemical and physical properties such as organic carbon (SOC), sand, silt, clay, and calcium carbonate ($CaCO_3$) contents in a sample site of southern Italy.

Spectral curves showed that the soils could be spectrally separable on the basis of chemical and physical properties. PLSR calibration models were derived for each of the soil properties and were validated with an independent data set. The optimum number of factors to be retained in the calibration models was determined by leave-one-out cross-validation. The accuracy of the calibration and validation models for the different soil properties was evaluated with the coefficient of determination (R^2) and the root mean squared error (*RMSE*). The results showed that predictions were satisfactory for all soil properties analyzed with high values of $R^2 > 80$.

A combination of Vis-NIR spectroscopy and multivariate statistical techniques, therefore, can be used as a rapid, low cost and quantitative means of characterizing the soils of southern Italy.

Keywords: Soil properties, Vis-NIR reflectance spectroscopy, PLSR, Southern Italy.

1. INTRODUCTION

Soil is one of the most important natural resources because it plays a key role in biochemical and geochemical cycling, water partitioning (storage and release), land protection and buffering, and energy partitioning, all of which are essential for supporting ecosystems [1]. Moreover, soil represents the largest pool of carbon (C), storing approximately 1500 PgC in the top 1 m [2, 3]; hence, even relatively small changes in soil C storage per unit area could have a significant impact on the global carbon balance. Therefore, accurately quantifying soil properties and their spatial and temporal variability is an important issue for sustainable land management, precision agriculture and soil mapping as well as for carbon sequestration. However, conventional methods of estimating soil properties, based on field or laboratory methods, are relatively complex, time consuming, and expensive when large numbers of soil samples need to be analyzed [4]. To overcome this, visible and near-infrared spectroscopy (Vis-NIR, 350 - 2500 nm) has become, in recent decades, an important tool for quantitative evaluation of soil properties, *e.g.*, carbonate content, organic carbon (OC), total nitrogen (N), iron (Fe) oxide minerals, soil texture [5-10]. Compared to conventional laboratory methods, the Vis-NIR spectroscopy technique has been accepted as rapid and cost effective, requiring minimal sample preparation and no hazardous chemicals. It is non-destructive and several soil properties can be determined from a single measure [*e.g.* 4, 10-15]. The Vis-NIR spectroscopy method is based on the simple assumption that the soil reflectance in the 350-2500 nm spectral region is a linear combination of the spectral signatures of its various components weighted by their abundance [16, 17]. So, small changes in physical, chemical, mineralogical and biological soil properties produce different spectral characteristics that can be identified by reflectance spectroscopy [12, 18-23].

Although measuring soil Vis-NIR reflectance requires only a few seconds, the reflectance spectra are largely non-specific due to interference resulting from the overlapping spectra of soil constituents that are themselves varied and interrelated [24]. Therefore, the physical, chemical and mineralogical properties of soils can be correlated to reflectance spectra by suitable multivariate calibration procedures [15, 25, 26] such as multiple linear regression (MLR), principal components regression (PCR), partial least-squares regression (PLSR) and artificial neural networks (ANN) [*e.g.* 6, 7, 12, 14, 21, 27-29].

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Several studies have successfully shown the effectiveness of Vis-NIR spectroscopy in determining the physical, chemical and mineralogical properties of soils of various pedo-climatic environments around the world. Yet few studies have been carried out in southern Italy [9, 10, 22, 23, 30, 31].

The objective of the paper was to assess the potential of Vis-NIR spectroscopy to determine various soil chemical and physical properties such as organic carbon (SOC), sand, silt, clay, and calcium carbonate ($CaCO_3$) contents in a sample area of southern Italy. To this end calibration models based on laboratory Vis-NIR spectroscopy coupled with PLSR analysis were developed.

2. MATERIAL AND METHODS

2.1. Description of the Study Area

The study area was the Turbolo watershed, with a surface of about 30 km^2, located in northern Calabria (southern Italy) between 39°32’25’’N and 39°29’51’’N latitude, 16°12’57’’E and 16°05’21’’E longitude (Figure **1**). Elevation ranges from 75 to 1,015 meters above sea level, with an average value of 292 m. Slope ranges between 0° to about 66° with a mean slope of 16°.

The climate is sub-humid, with a mean annual precipitation of 1,200 mm, distributed over 105 days. Rainfall peaks occur in the period from October-March, which accounts for more than 60% of the precipitation [32]. The mean annual temperature is 16°C, and mean monthly temperatures range between 7°C in January and 26°C in August.

The Turbolo watershed in the western sector is dominated by a mountainous landscape characterized by steep slopes shaped on Palaeozoic metamorphic rocks (mainly gneiss and schist), intensely fractured and weathered, and in many places covered by thick regolith. In the eastern sector, the topography is characterized by gentle slopes and fluvial terraces cut on sedimentary terrains of Neogene-Quaternary ages

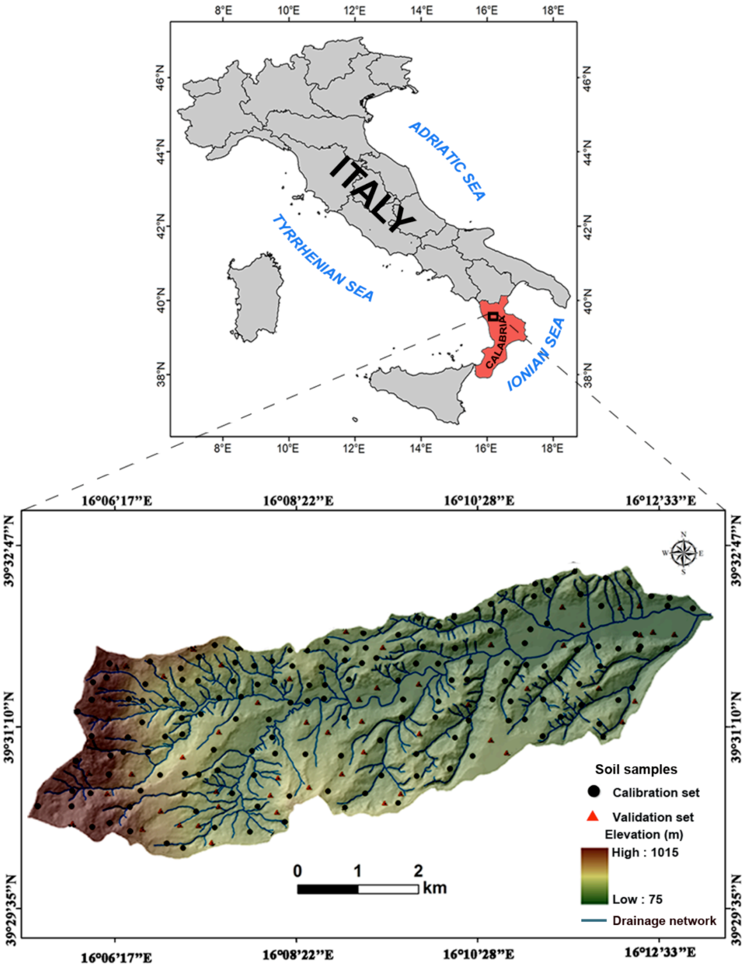

Figure 1: Study area and soil sampling locations.

consisting mostly of clays, sands and conglomerates [33]. Alluvial and colluvial deposits are present along the valley floors [34].

The distribution of soil types is closely related to the geological, geomorphologic and climatic properties of the area. According to the pedological data, derived from the soil map of Calabria [35] based on Soil Taxonomy [36], the dominant soils types are Alfisols, Inceptisols and Entisols. In addition, on clayey hillsides, soils exhibit prominent vertic properties, with a high structural dynamism characterized by the development of cracks (due to shrinkage of desiccated clays) on the surface in the dry season, subsequently closed by water infiltration, and consequent swelling of clays in the following wet season. The pedoclimatic regime is xeric and thermic, shifting to udic and mesic in the mountainous parts [35]. The prevailing soil textural classes are sandy loam and sandy clay loam [34, 37]. The soil profiles frequently appear truncated or severely degraded by water erosion and mass wasting [38, 39].

Regarding the land use, about half of the study area is agricultural, mainly cropland and olive groves [34], whereas more than 20% has a shrubby and herbaceous cover often left to pasture. The remainder consists of woodland, which is widespread in the western sector of the watershed.

2.2. Sampling and Analysis of the Soil

A set of 215 georeferenced surface soil samples (about 0-20 cm depth) were collected within the study area, by means of an auger sampler (Figure **1**). The sampling sites were carefully selected on the basis of the most representative soil-landscape features, such as geological substrate, topographic characteristics, soil types, land use/cover and development/ degradation conditions of the topsoil. The locations of the sampling sites were recorded with a Garmin eTrex30 GPS, with an accuracy of 3-5 m.

The samples were brought to the laboratory, air dried, gently crushed in an agate mortar to break up larger aggregates and visible roots were removed; afterwards each sample was sieved at 2 mm (fine earth fraction), homogenized and quartered and then split into two sub-samples. One was used to determine certain soil properties (organic carbon, calcium carbonate, clay, silt and sand contents) using conventional laboratory methods, and the other one for the spectroscopic measures.

The physical and chemical properties were determined according to the Italian Official Methods for Soil Analysis [40, 41]. The relative proportion of sand (2-0.05 mm), silt (0.05–0.002 mm), and clay (less than 0.002 mm) content was determined through the hydrometer method, after pre-treatment with sodium hexametaphosphate as a dispersant. The content of soil organic carbon (SOC) was determined by Walkley-Black method and calcium carbonate content ($CaCO_3$) was determined by the pressure calcimeter method.

2.3. Soil Vis-NIR Reflectance Measurements

Soil Vis-NIR reflectance was measured in the laboratory, under artificial light, using an ASD FieldSpec IV spectroradiometer (Analytical Spectral Devices Inc., Boulder, Colorado, USA), which combines three spectrometers to cover the portion of the spectrum between 350 and 2500 nm, with a sampling interval 1.4 nm for the 350 - 1000 nm region and 2 nm for the 1000 - 2500 nm region. The spectroradiometer was located in a nadir position at a distance of 10 cm from the sample, allowing for radiance measurements within a circular area of approximately 4.5-cm diameter. For the spectral measurement, each soil sample was placed inside a circular black capsule of 10 cm in diameter and 1 cm depth and levelled with a spatula to obtain a smooth surface. A 50 Watts halogen lamp with a zenith angle of 30°, located at a distance of approximately 0.25 m from the soil sample was used as a light source. The measured radiance was transformed to spectral reflectance as the ratio between the radiance reflected by the soil and that of a standard white reference plate (Spectralon) measured under the same illumination conditions. The noise level in the spectral signal was reduced by averaging 50 spectra for each soil sample. In addition, to eliminate any possible spectral anomalies due to geometry of measurement, four replicate scans were acquired and averaged for every soil sample by rotating the sample by 90° (Figure **2**).

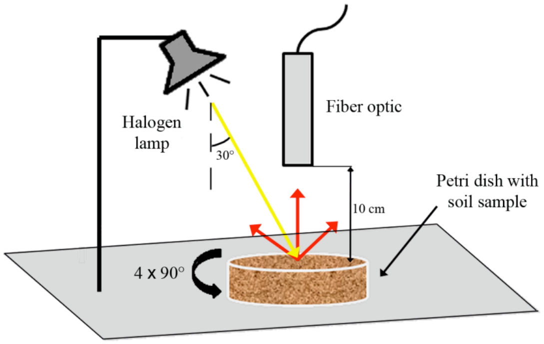

Figure 2: Setup of the experimental equipment for soil Vis-NIR spectral measurements.

Finally, the average reflectance curves were resampled at 10-nm interval, to reduce spectral dimensionality.

2.4. Calibration and Validation Models

In order to develop models based on spectra and reference laboratory data of the soil samples, the multivariate statistical procedure was used (Figure **3**). Prior to developing the spectra calibration models, therefore, pre-treatment was carried out to minimize noise and optimize calibration accuracy. The measured Vis-NIR reflectance (R) spectra were transformed to apparent absorbance through log(1/R) to enhance the linearity between the measured absorbance and the values of the relevant soil properties. The absorbance spectra were mean-centred to ensure that all results would be interpretable in terms of variation around the mean; then they were smoothed through a median filter algorithm with a first derivative to remove an additive baseline [42]. Finally, multiplicative signal correction (MSC) was applied to the absorbance spectra to correct for light scattering variations [43]. Details on pre-processing methods can be found in Martens and Næs [25] and in Næs *et al*. [44].

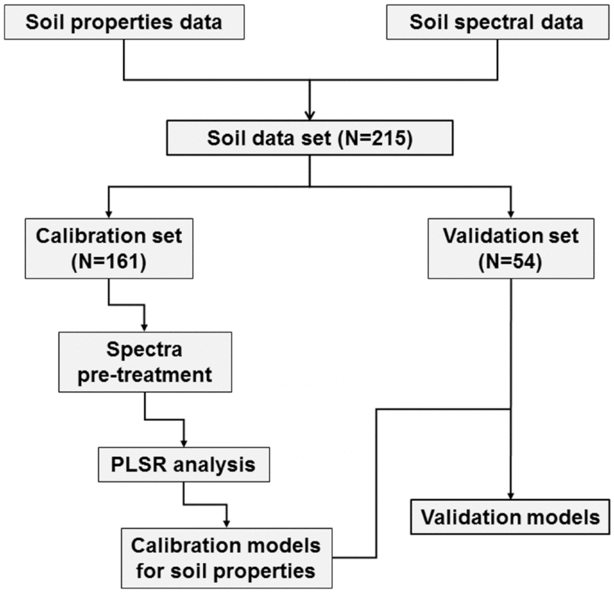

Figure 3: Flow chart of the procedure used to develop Vis-NIR PLSR prediction models for the determination of soil properties.

Partial least squares regression (PLSR) [14, 25], was chosen from the available multivariate statistical methods to determine the relation between absorbance and soil properties (clay, silt, sand, SOC and $CaCO_3$) determined by conventional methods. Pre-treatment of data and the PLSR analysis were performed with PArLeS vs3.1 software developed by Viscarra Rossel [42]. The idea behind PLSR is to find a few linear combinations (components or factors) of the original **X**-values (spectral data) and to use only these combinations in the regression equation [44]. In this way, the irrelevant and unstable information is discarded and only the most relevant part of the **X**-variation is used for regression; thus, the problem of collinearity is solved and more stable regression equations obtained [44]. PLSR reduces the Vis-NIR matrix to a small number of statistically significant components and is based on the latent variable decomposition of two sets of variables: the set **X** of predictors (matrix $n \times \mathbf{N}$, where n is the number of observations and **N** is the number of wavelengths) and the set **y** of response variable (vector $n \times 1$ of soil properties). The latent variables, which are orthogonal factors that maximize the covariance between independent (**X**) and dependent variables (**y**), explain most of the variation in both predictors and responses. The optimal number of latent variables was chosen through a one-at-a-time cross-validation as the number that minimizes the predicted residual sum of squares.

To evaluate the accuracy of the PLSR regression models the dataset was separated randomly into two subsets: calibration set (75%, n=161) for developing the prediction model and validation set (25%, n=54) to test the model's accuracy (Figures **1** and **3**).

The leave-one-out cross-validation procedure was used to test the predictive significance of each PLSR component and determine the number of factors (latent variables) to be retained in the calibration model: one sample was left out of the global data set and the model was calculated on the remaining data points. The value for the left-out sample was then predicted and the residual computed. The process was repeated with another sample from the data set, and so on, until every sample had been left out once. The predictive ability of the cross-validation models was evaluated by the coefficient of determination (R^2_{Cal}) and the root mean square error of calibration ($RMSE_{Cal}$).

The models were independently validated through the validation set, and the coefficient of determination (R^2_{Val}) and the root mean square error of validation ($RMSE_{Val}$) were computed to check the goodness of prediction.

3. RESULTS AND DISCUSSION

The summary statistics of the soil properties for the exhaustive, calibration and validation data sets are

Table 1: Summary Statistics of the Exhaustive (*E*), Calibration (*Cal*) and Validation (*Val*) Data Sets of Soil Properties

	SOC			Sand			Silt			Clay			$CaCO_3$		
Samples	***E***	***Cal***	***Val***	***E***	***Cal***	***Val***	***E***	***Cal***	***Val***	***E***	***Cal***	***Val***	***E***	***Cal***	***Val***
Number	215	161	54	215	161	54	215	161	54	215	161	54	215	161	54
Min (%)	0.2	0.2	0.2	3.0	4.0	3.0	1.0	1.0	6.0	4.0	4.0	4.0	0.0	0.0	0.0
Max (%)	7.0	7.0	6.2	93.0	93.0	79.0	58.6	53.0	58.6	66.0	54.0	66.0	28.4	28.4	25.3
Mean (%)	1.7	1.7	1.8	59.5	60.7	55.9	20.6	19.5	23.9	19.9	19.7	20.4	6.0	5.9	6.2
St. Dev. (%)	1.2	1.2	1.2	17.9	17.8	18.0	12.5	12.5	12.0	10.3	10.0	11.3	6.5	6.3	7.0
Median (%)	1.5	1.4	1.7	62.0	63.0	59.5	19.0	17.0	23.0	18.0	18.0	18.0	2.8	2.8	2.5
Skewness (-)	1.6	1.6	1.3	-0.8	-0.7	-1.1	0.7	0.7	0.8	1.1	0.8	1.6	1.1	1.0	1.1
Kurtosis (-)	3.0	3.2	2.2	0.6	0.4	1.0	-0.1	-0.3	0.4	1.8	0.2	4.0	0.3	0.2	0.2

reported in Table **1**. The content of SOC of the exhaustive data set ranges from a minimum of 0.2 % to a maximum of 7.0 % with a mean value of 1.7 %, indicating that most topsoil samples have a moderate SOC content.

The sand content for the exhaustive data set ranges from 3.0 % to 93.0 % with a mean value of 59.5 % (Table **1**). The silt content varies from 1.0 % to 58.6 % with a mean value of 20.6 %, while clay content ranges from 4.0 % to 66.0 % with a mean value of 19.9 %. The data shows that most soil samples have a moderate to high sand content and are generally poor in clay (Table **1**); the soils, therefore, can be mainly classified as loamy sand, sandy loam and sandy clay loam.

$CaCO_3$ content ranges from 0.0 to 28.4 % with mean value of 6.0 % (Table **1**). Soil samples with high contents of $CaCO_3$ and sand, together with low SOC indicate that many soils are poorly developed and/or have undergone considerable erosion [22, 37].

Descriptive statistics of soil properties of the calibration set and the validation set (Table **1**) are quite similar to those of the exhaustive data set. In addition, all soil properties have an asymmetric distribution as shown by the values of skewness (Table **1**).

The spectral curves of all soil samples are plotted in Figure **4a** and show a typical pattern in each wavelength domain; in particular, reflectance is generally lower in the visible range (350-700 nm) and higher in the near infrared range (700-2500 nm).

In accordance with Demattê *et al*. [45], soils are spectrally separable on the basis of physical, chemical and mineralogical composition or molecular groups. Their spectral curves, therefore, showed variation in shape, overall reflectance intensity and absorption bands (depth and amplitude) around 1400, 1900 and 2200 nm (Figure **4a**). These bands can be associated with clay minerals, OH features of free water at 1400 and 1900 nm, and lattice OH features at 1400 and 2200 nm [14, 16]. In addition, the spectra showed a small reflectance peak around 2200 nm, which may be due to organic molecules (e.g. CH_2, CH_3, and NH_3), Si\OH bonds, cation\OH bonds in phyllosilicate minerals (e.g. kaolinite, montmorillonite) [46]. In some soil spectra, the absorption band at 2200 nm is associated with a smaller band (doublet) at 2160 nm, due the combination of the OH-bond fundamental stretch with the Al-OH fundamental bending mode [47].

Figure **4b** shows the comparison of the spectral curves of soils with different values of particle size distribution, SOC and $CaCO_3$ content. In accordance with other studies [*e.g.* 5, 10, 12, 48], SOC is a property which has a strong influence on spectral response. Reflectance intensity was relatively high, throughout the 350 nm to 2500 nm wavelength range, for soils with low content of SOC. Spectral curves with low reflectance show a concave or linear shape, which results in a decrease of curve slope between 500 and 800 nm domain (Figure **4b**), that is typical of soils with high SOC content [49].

Moreover, variations in soil particle size distribution modified the shape and overall reflectance intensity of the soil spectra [23]. In fact, it was observed that reflectance intensity, in the entire Vis–NIR spectrum domain, is relatively high for soils with loamy sand texture with over 70 % sand content (Figure **4b**); this was probably due to the high amount of quartz in the sand fraction, which raised the intensity of spectral reflectance [50, 51]. Conversely, the soil reflectance decreased when clay content, dominated by

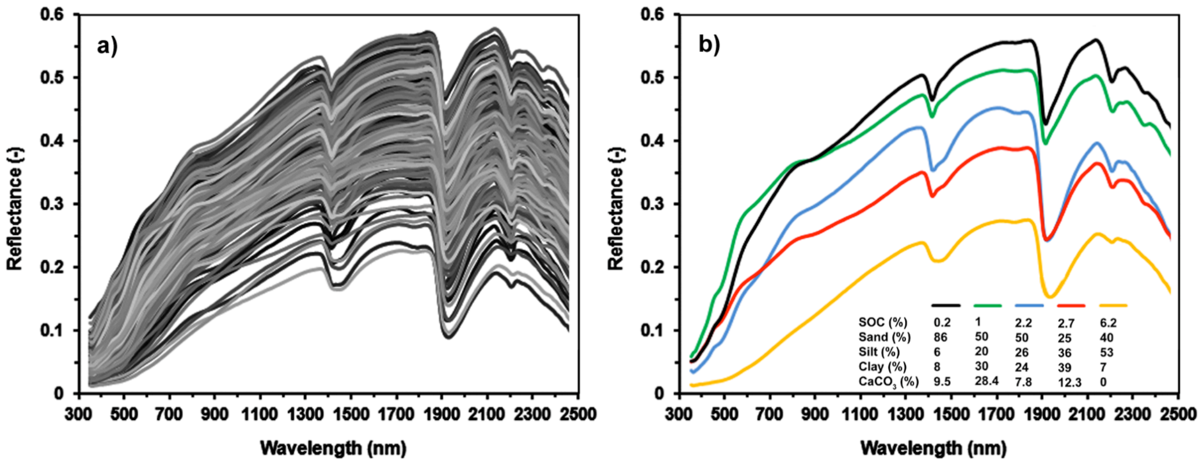

Figure 4: **a**) Spectral curves of all soil samples; **b**) Spectral curves selected to illustrate the variation of soils properties in the study area.

phyllosilicates, increased [52, 53] and, consequently, the SOC content increased [10, 54].

Finally, in many soil spectra an absorption band around a wavelength of 2342 nm was observed (Figure **4b**), which is indicative of the presence of $CaCO_3$ in the soil [55]. Moreover, it was generally the case that soil reflectance increases with $CaCO_3$ content (Figure **4b**).

Cross-validation results of the PLSR prediction model, obtained from the relationships between laboratory Vis-NIR spectra and soil properties, including SOC, sand, silt, clay and $CaCO_3$, of the calibration set are shown in Figure **5a**. The PLSR results show that calibration models fully account for the variance in each soil property.

The optimum number of factors used in the calibration models, determined on the basis of the best cross validation results (lowest *RMSE* and the highest R^2), were 10 for SOC, 12 for sand, 16 for silt, 11 for clay and 13 for $CaCO_3$ (Figure **5a**). A very good calibration was obtained for all soil properties with R^2 greater than or equal to 0.80. The high accuracy of the calibration models was also indicated by the lowest *RMSE* of cross-validation.

The best results were obtained for $CaCO_3$ and SOC, with a *RMSE* of 1.93 % and 0.41 % and R^2 of 0.91 and 0.88, respectively (Figure **5a**). The estimation obtained for silt (R^2 = 0.80; *RMSE* = 5.69) was less accurate.

The PLSR models built in this study from Vis-NIR reflectance compare well with the results obtained by other researchers [9, 10, 14, 15, 31, 54, 56, 57, 58 and many others].

Moreover, the predictive power of the calibration model of each soil property was tested against an independent data set (validation set); the scatter plots of the validation models were reported in Figure **5b**. The results obtained were very good for SOC, sand, clay and $CaCO_3$ with R^2 greater than or equal to 0.80, while the results for silt were less satisfactory (R^2=0.77). In addition, these results are in close agreement with the results obtained from the PLSR cross-validation models.

4. CONCLUSIONS

In this study soil Vis–NIR spectroscopy and PLSR were used to determine SOC, sand, silt, clay, and $CaCO_3$ content in an area of southern Italy.

Reflectance spectroscopy allowed for the detection of soil changes through variations in reflectance intensity, absorption features and spectral shape. Visual inspection of the spectral curves demonstrated that SOC content and particle size distribution have an important influence on spectral reflectance in the Vis-NIR domain. Moreover, comparison of spectral curves showed that soil samples could be spectrally separable on the basis of soil properties; in particular, reflectance intensity was observed to be relatively higher for soils with low SOC content, high $CaCO_3$ content and loamy-sand texture, which have a high sand content.

The results show that the proposed approach is suitable for predicting the soil properties considered in this study. The results of cross validation for the calibration models indicated a very good fit. Values of R^2 ranged between a minimum of 0.80 for silt and a

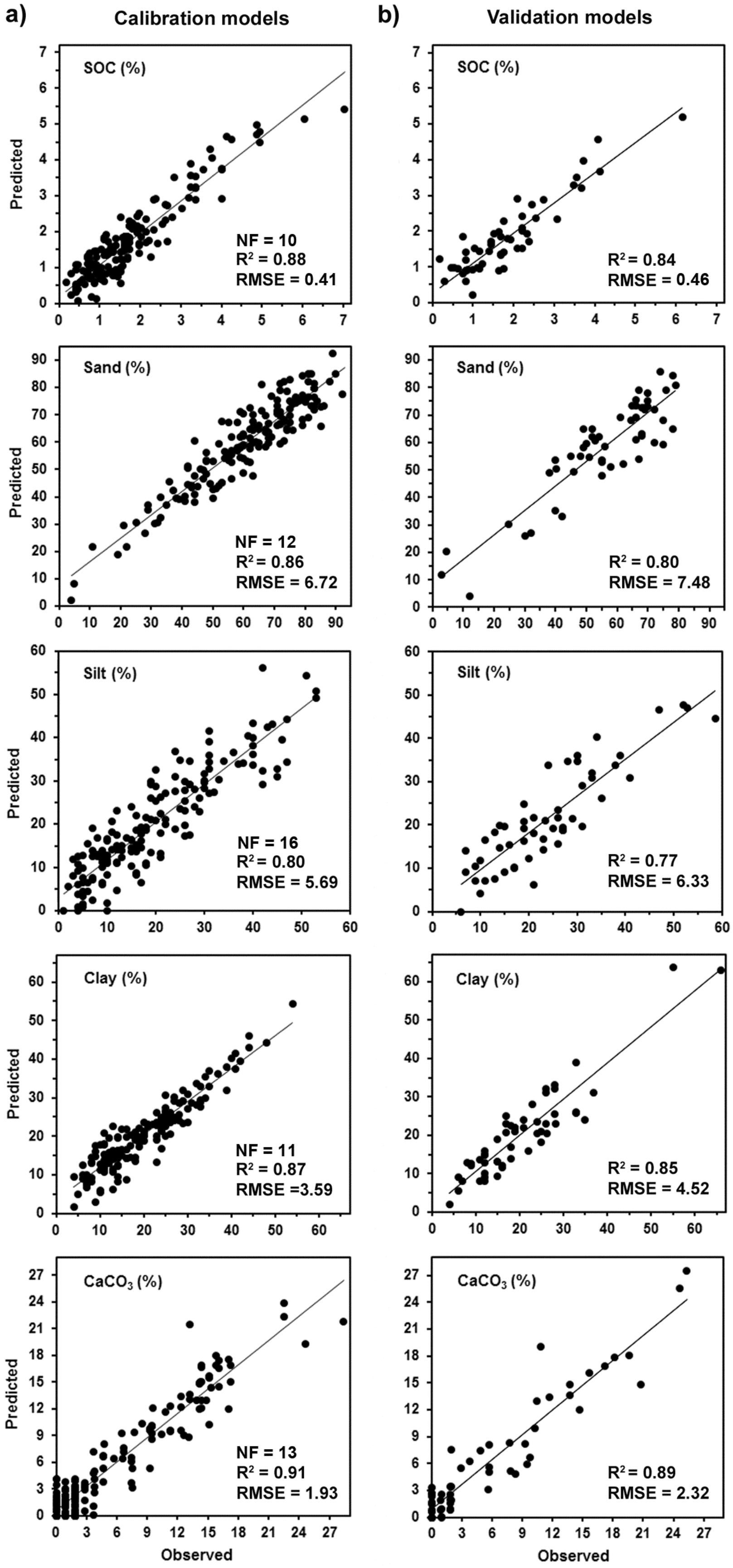

Figure 5: Scatter-plots of observed versus predicted soil properties for calibration models (**a**) and validation models (**b**). NF = number of factors; R^2: coefficient of determination; *RMSE*: root mean square error.

maximum of 0.91 for $CaCO_3$ content. The results for the validation models were also satisfactory and in accord with those obtained by other authors.

Finally, the approach proposed in this study could be extended to soil surveys in other areas of the Calabria region.

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