

Production of an innovative biowaste-derived fertilizer: rapid monitoring of physical-chemical parameters by hyperspectral imaging

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ABSTRACT

In this study the possibility to apply HyperSpectral Imaging (HSI) as a fast and non-destructive technique for the characterization of an innovative biowaste-derived fertilizer was explored. Different mixtures of urban organic waste, farm organic residues, biochar and vegetable active principles were selected and utilized in two different European countries, Italy and Spain, for the production at pilot scale plant of the innovative fertilizer. The biowaste-derived fertilizer samples were collected from the piles at different curing time and acquired by hyperspectral imaging in the near infrared wavelength range (1000-1700 nm). Classical physico-chemical analyses were carried out on the same samples in order to find correlations between the measured parameters and the acquired reflectance spectra. The investigated parameters were: pH, soluble total organic carbon and soluble total nitrogen.

Hyperspectral data were processed adopting chemometric strategies through the application of principal component analysis, for exploratory purposes, and partial least squares analysis to build the correlation models between spectral features and measured physico-chemical parameters. Good correlations were found for all the investigated parameters. Results show as the proposed approach, based on hyperspectral imaging, is suitable to be adopted for a rapid and non-destructive fertilizer production monitoring.

Keywords: biowaste recycling, fertilizer production, hyperspectral imaging, near infrared spectroscopy.

1. INTRODUCTION

The world market of chemical fertilizers is extremely large, being currently responsible for 50% of the world's food supply [1]. During the 2013/2014 European growing season, fertilizers containing about 10.7 Mt of nitrogen (N), 2.5 Mt of phosphate (P₂O₅) and 2.7 Mt of potash (K₂O) were applied to 133.5 million hectares of farmland in the 27 EU countries. By 2023/2024, forecasters expect annual nitrogen, phosphate and potash fertilizer consumption to reach 10.8 Mt, 2.7 Mt and 3.0 Mt, respectively. Unfortunately, chemical fertilizers have an heavy impact on the environment, both concerning pollution and consumption of non-renewable resources [2]. Fertilizer production is also a highly energy demanding process, requiring significant amounts of fossil fuels.

The concept of circular economy has started to be applied also to the fertilizer industry, closing the “fertilizer loop”, by developing strategies for more nutrients recycling, reducing at the same time pollution and energy consumption. At the farm level, the attention is mainly focused on three aspects: i) crop waste composting, ii) anaerobic digestion of animal slurries for energy generation and iii) a more efficient integration of organic waste into the overall fertilization strategy.

The use of composted organic wastes to improve soil structure and fertility, as well as plant growth, has been widely investigated in the last decades and is of growing interest [3-6]. In this perspective, the production monitoring of a reduced salinity fertilizer obtained from different organic wastes is introduced in this paper, utilizing hyperspectral imaging, an innovative sensor-based technique. The work was carried out in the framework of the LIFE RESAFE Project (LIFE12 ENV/IT/000356) “*Innovative fertilizer from urban waste, bio-char and farm residues as substitute of chemical fertilizers*”. The main objective of the RESAFE project was to demonstrate the production and use of a fertilizer characterized by reduced salinity, obtained from Urban Organic Waste (UOW), Bio-Char (BC), Farm Organic Residues (FOR) mixed with Vegetable Active Principles (VAP) [7]. The fertilizer was preliminary produced at laboratory scale to select the best “recipe” of mixed organic waste (i.e. quality and quantity of raw materials) [8] and

then at pilot scale plant to be tested in agricultural applications.

The biowaste-derived fertilizer production process performed at laboratory scale was monitored using an innovative sensing technique, based on hyperspectral imaging (HSI) working in the near infrared (NIR) range (1000-1700 nm) [9]. The **HyperSpectral Imaging** (HSI) based detection and control strategies set up at laboratory scale have been then systematically applied at pilot plant scale.

HSI combines classical spectroscopy and image analysis in order to simultaneously obtain sample spectral and spatial information, producing for each pixel of the acquired image a full spectrum constituted by all the wavelengths belonging to the investigated spectral range, VIS, NIR or SWIR [10-11]. Such emerging technique has many advantages, being non-invasive, non-destructive, non-polluting, fast and relatively cheap.

The use of HSI is rapidly growing in the last years in many sectors, including agriculture, with applications related to vegetation mapping, crop disease, stress and yield detection, component identification in plants and detection of impurities [12-14]. Concerning soils, the attention was mainly focused on the prediction of nutrient contents, pH, humidity, organic matter, etc. [15]. This is possible thanks to the detection and characterization of the spectral signature that is influenced by the overtones and combinations of molecular vibrations of the functional groups C-H, O-H, N-H [16].

Main aim of this study was thus to predict the values of the physical-chemical parameters affecting the “evolution” different organic waste derived products (i.e. innovative fertilizers) following an HSI based approach in the NIR range, in order to set-up investigation strategies addressed to perform a fast, low cost, robust and reliable detection of those parameters affecting fertilizer evolution/maturation, as: pH, soluble carbon and soluble nitrogen. The determination of the same parameters following the classical approach is, in fact, laborious, expensive and time-consuming.

2. MATERIALS AND METHODS

2.1 Investigated samples

Different organic wastes (i.e. UOW, FOR and BC) were selected and used in order to produce the RESAFE fertilizer in two different countries: Italy and Spain. For the Italian fertilizer recipe, UOW comes from recycled kitchen waste, collected by private citizens or by collective and commercial structures, as well as from agro-industrial activities; FOR consists of dried poultry manure and BC is a charcoal, specifically obtained from the biomass pyrolysis. In the Spanish fertilizer recipe, UOW comes from an industrial composting process, characterized by a medium level of degradation; FOR consists of fresh horse manure and BC, not degradable and stable, is of industrial origin. In both countries the biowastes were mixed with VAP, an innovative vegetable complex enzymatic blend prepared from selected plants [17], developed with the main aim to accelerate the process of aerobic stabilization of biomass and already tested in different applications [8].

In order to produce the fertilizer at pilot scale plant, both in Italy and Spain three different heaps were realized using in total 6690 kg of biowaste (2230 kg for each heap). In Italy the proportion of materials used for each heap was: 1000 kg (50%) FOR + 1000 kg (50%) UOW + 200 kg (10%) BC + 30 kg VAP, whereas in Spain were: 800 kg (40%) FOR + 1200 kg (60%) UOW + 200 kg (10%) BC + 30 kg VAP.

At the base of each heap, a layer of 40 cm of waste mixture was placed and the VAP was put in the center of the pile. A mixture of UOW, FOR and BC was then utilized to cover the whole layer. The coverage was about 50 cm thick. Furthermore, in order to reduce heap breathability and water losses a layer of compost characterized by a fine texture was utilized to realize the final heat coverage. Finally the three heaps were covered by a plastic tunnel to maintain heap optimal humidity and temperature conditions. The curing time for the fertilizer production was set at 90 days.

For each heap, three samples were collected at the beginning (T=0) and at the end (T=90 days) of the curing process, for a total of 18 samples in Italy and 18 samples in Spain.

2.2 Analytical methods

Classical chemical analyses and hyperspectral imaging were carried out on the investigated Italian and Spanish samples, in order to correlate the chemical parameters and the spectral signatures of the samples.

2.2.1 Chemical analyses

Chemical analyses were carried out at the Laboratory of CEBAS-CSIC (Department of Soil and Water Conservation and Organic Wastes Management, Murcia, Spain). The measured parameters utilized to find correlation with the

hyperspectral imaging data were pH, Soluble Total Organic Carbon (STOC) and Soluble Total Nitrogen (STN). pH was determined using a pH-meter in a sample solution of 1:5 in distilled water (Figure 1a). STOC and STN were extracted by shaking for 2h a mixture of samples (Figure 1b) and distilled water, at a 1:5 solid/liquid ratio, and then measured in a AnalytkJena Multi N/C 3100 Analyzer (Figure 1c), after centrifuging and filtering utilizing ash-less filter paper.

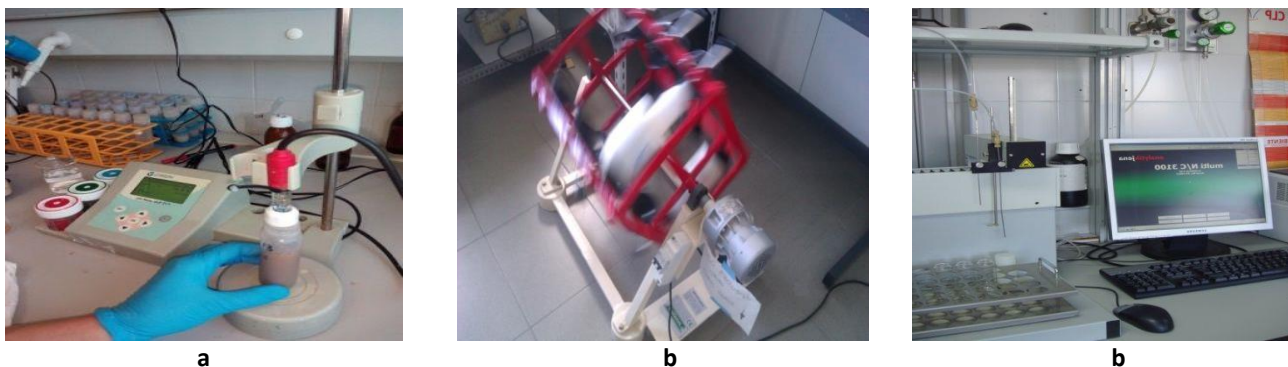


Fig.1 Image sequence showing the different devices/procedures utilized to perform pH, Soluble Total Organic Carbon (STOC) and Soluble Total nitrogen determination (STN). a: pH measurement, b: sample shaking and c: STOC and STN analyses performed by AnalytkJena Multi N/C Analyzer

2.2.2 Hyperspectral imaging analyses

Architecture set-up

Hyperspectral data were acquired at laboratory of DICMA (Sapienza University, Rome, Italy) using a NIR Spectral Camera™ (Specim, Finland) equipped with an ImSpector N17E™ imaging spectrograph working with a spectral sampling/pixel of 2.6 nm, coupled with a Te-cooled InGaAs photodiode array sensor and a diffuse light cylinder device with an internal aluminum coating (Figure 2). In order to provide the required energy for the process, the cylinder contains five halogen lamps producing a continuous spectrum signal optimized for spectra acquisition in the NIR wavelength range (1000-1700 nm). The device works as a push-broom type line scan camera, allowing the acquisition of spectral information for each pixel in the line. Black and white calibration was automatically performed. The transmission diffraction grating and optics provide high light throughput and high quality and distortion-less image for the device. The result of acquisition is constituted by a digital image where each column represents the discrete spectrum values of the corresponding element of the sensitive linear array.

Spectral data analysis: spectra preprocessing, data exploration and modelling

Spectral data analysis was carried out by standard chemometric methods [11,18] utilizing the PLS Toolbox (Version 7.9.2, Eigenvector Research Inc.) running inside Matlab® (Version 8.4.0, The Mathworks Inc.).

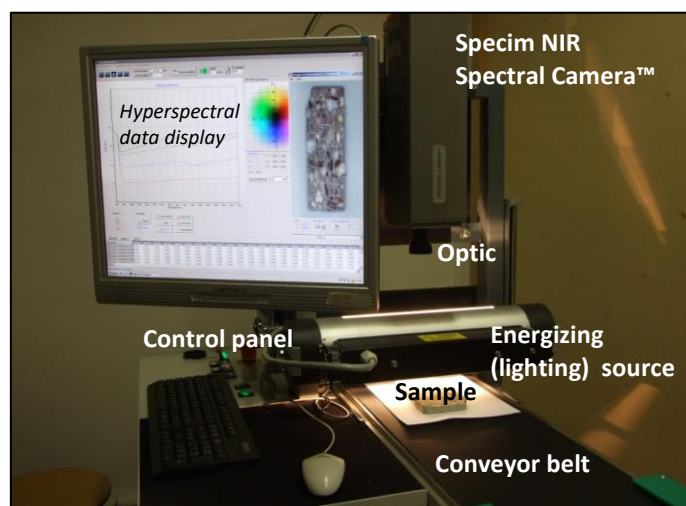


Fig. 2 Overall view of the **HyperSpectral Imaging** (HSI) based acquisition/detection architecture

In order to eliminate unwanted effects due to lighting/background noise raw spectra were preliminary cut, at the beginning and at the end of the investigated wavelength range. The wavelength reduction was from 121 to 95, with a resulting investigated spectral range between 1000 and 1650 nm. The following pre-processing algorithms were sequentially applied to highlight spectral features of the different samples: *Smoothing*, *2nd Derivative* and *Mean Centering* (MC) [19].

Smoothing algorithms are often used to remove high frequency signals, which are characteristic of noise, thereby enhancing chemical group signals. Derivatives are methods usually adopted to remove unimportant baseline signal from samples by taking the derivative of the measured responses with respect to the wavelength. MC represents one of the most utilized preprocessing approaches. It computes the mean of each column and subtracts this from the column.

PCA was applied to the preprocessed hyperspectral images for exploratory purposes. It is a powerful and versatile method capable of providing an overview of complex multivariate data. PCA can be used e.g. for revealing relations between variables and relations between samples, detecting outliers, finding and quantifying patterns, generating new hypotheses as well as many other things [20]. PCA compresses the data by projecting the samples into a low dimensional subspace, whose axes (i.e. the principal components, PCs) point in the directions of maximal variance.

In order to evaluate possible correlations between spectral responses and measured physical-chemical parameters, **Partial Least Squares** (PLS) was then applied. PLS is an efficient tool already tested for the prediction of different nutrients and other important parameters in agricultural applications [21-24]. PLS projects the original data into a low-dimensional space formed by a set of orthogonal latent variables by a simultaneous decomposition of X (spectral matrix) and Y (elemental concentration matrix) that maximizes the covariance between X and Y [25].

The evaluation of the calibration performance is estimated by computing the squared correlation coefficient (r^2), the **Root Mean Squared Error of Calibration** (RMSEC) and **Root Mean Squared Error of Validation** (RMSECV) and the number of **Latent Values** (LVs).

The calibration model was chosen considering the PLS Latent Variables (LVs) that minimize the value of the RMSECV, and the higher validation correlation coefficient r^2 . In particular, r^2 is the correlation coefficient between the estimated and predicted or calibrated values and is represented by the following expression:

$$r^2 = \left(\frac{\sum_{i=1}^N (C_i - C'_i)^2}{\sum_{i=1}^N (C'_i - C_m)^2} \right) \quad (1)$$

where C_i is the known value, C'_i is the calculated value by the calibration equation, C_m is the mean of the value of all samples in the calibration set. The equation utilized to evaluate RMSEC is:

$$RMSEC = \sqrt{\left(\frac{\sum_{i=1}^N (C_i - C'_i)^2}{N - 1 - p} \right)} \quad (2)$$

where C_i is the known value, C'_i is the calculated value by the calibration equation, N the number of samples and p is the number of independent variables in the regression. The equation utilized to evaluate RMSECV is:

$$RMSECV = \sqrt{\left(\frac{\sum_{i=1}^N (C_i - C'_i)^2}{N} \right)} \quad (3)$$

with C_i , the parameter prediction value of the sample i in calibration data set, C'_i , the measured parameter value of sample i in calibration data set, N, the number of samples in calibration dataset.

3. EXPERIMENTAL RESULTS AND DISCUSSION

3.1 Chemical analyses results

The results of chemical analyses are reported in Table 1. In Italian and Spanish samples collected at the beginning of the

curing process (T=0 days), the observed pH values range between 8.00 and 8.43, indicating a basic value. Table 1. Results of the measured chemical parameters.

	pH		Soluble Total Organic Carbon (%)		Soluble Total Nitrogen (%)	
	T0	T90	T0	T90	T0	T90
Italy	8.20	9.49	3.02	1.64	0.82	0.51
	8.00	9.51	2.83	1.60	0.73	0.50
	8.07	9.49	2.53	1.64	0.87	0.53
	8.35	9.50	2.26	1.30	0.78	0.50
	8.28	9.53	2.43	1.32	0.82	0.50
	8.21	9.52	2.22	1.25	0.73	0.50
	8.10	9.53	2.79	2.59	0.87	0.51
	8.30	9.54	2.30	2.75	0.78	0.50
	8.32	9.52	2.53	2.65	0.81	0.58
Spain	8.22	8.65	1.98	1.20	0.28	0.20
	8.00	8.66	1.83	1.17	0.26	0.20
	8.22	8.74	1.92	1.18	0.28	0.21
	8.36	8.71	2.55	1.23	0.39	0.19
	8.42	8.70	2.25	1.20	0.33	0.19
	8.43	8.70	2.44	1.23	0.36	0.19
	8.34	8.71	2.28	1.25	0.32	0.19
	8.38	8.72	2.32	1.29	0.33	0.19
	8.34	8.71	2.25	1.25	0.32	0.19

At the end of the curing process (T=90 days), the pH value of the organic fertilizers increased in all samples, probably as a consequence of organic acids degradation. The achieved results (Table 1) are in agreement with those reported in the literature [26-28].

Soluble total organic carbon show values between 1.83 and 3.02 % before processing and values around 1.17-2.75% after processing. Soluble total nitrogen shows values between 0.26 and 0.87 % before processing and around 0.19 and 0.58% after processing, respectively. These values underlined that these parameters are degraded during process. It is also possible to see that for all the parameters, Italian samples showed higher values than Spanish samples.

These results are in good agreement with recipes characteristics. Poultry manure, utilized in Italian recipes, in fact, is characterized by higher contents of nitrogen and carbon than horse manure, utilized in the Spanish ones [29].

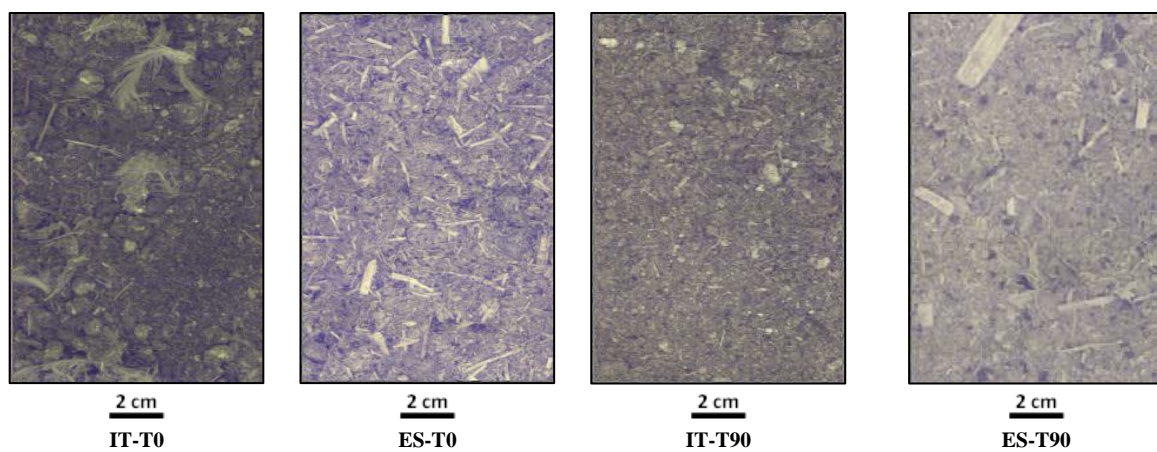
3.2 Sample spectral features and Principal Component Analysis (PCA)

For each acquired sample image, a region of interest was selected (Figure 3) and a mosaic image, embedding all the investigated spectral fertilizers products attributes, was created (Figure 4) for Italian and Spanish samples.

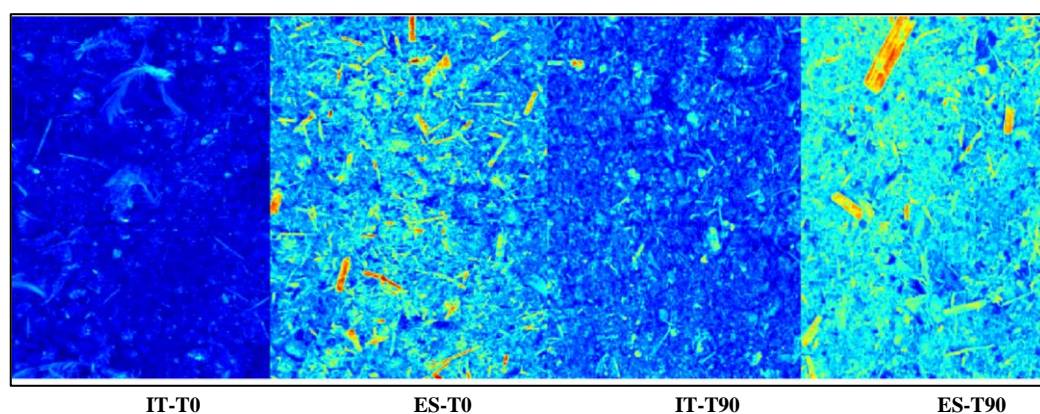
The average raw reflectance spectra and the corresponding preprocessed spectra are shown in Figure 4a and 4b, respectively. The average raw spectra show that at 90 days (T90) the reflectance is higher than at the beginning (T0). It is possible to see, also, that Spanish samples have higher reflectance than Italian ones, due to the different samples composition. In fact, poultry and horse manure have a different content of STOC, STN and pH that influence both spectral reflectance and shape.

In Figure 5 it is reported the PCA score plot (PC1-PC2) (Figure 5a) and the loading plot (Figure 5b). The score plot (Figure 5a) shows a good separation for all the analysed samples. The first two PCs account for 97.31% of the variability, PC1 providing 95.83% and PC2 1.48%.

At the beginning and at the end of the process, Italian samples shows negative values of PC1 and PC2, while Spanish sample at T=0 has positive value of PC1 and negative value of PC2. On the contrary, at T=90 Spanish sample shows negative value of PC1 and positive value of PC2.

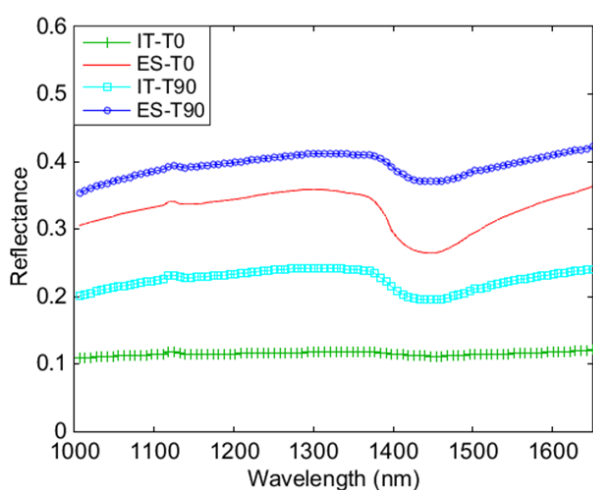


a

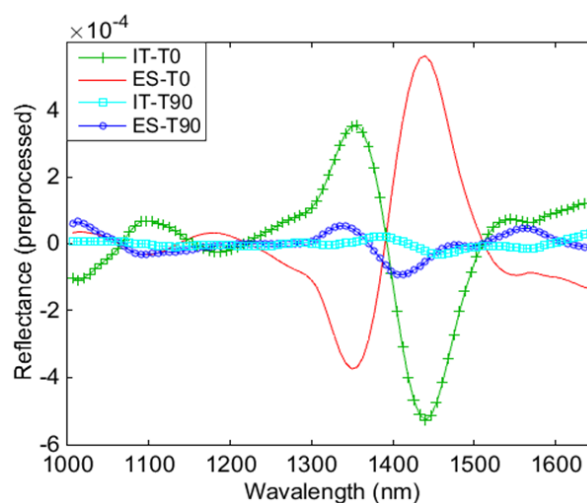


b

Fig. 3 Source image of Italian and Spanish innovative fertilizers samples collected at different curing time and corresponding hyperspectral mosaic image.



a



b

Fig. 4 Average raw (a) and preprocessed (b) reflectance spectra of waste-derived fertilizers. Preprocessing sequentially applied: *Smoothing*, *2nd Derivative* and *Mean Center (MC)*.

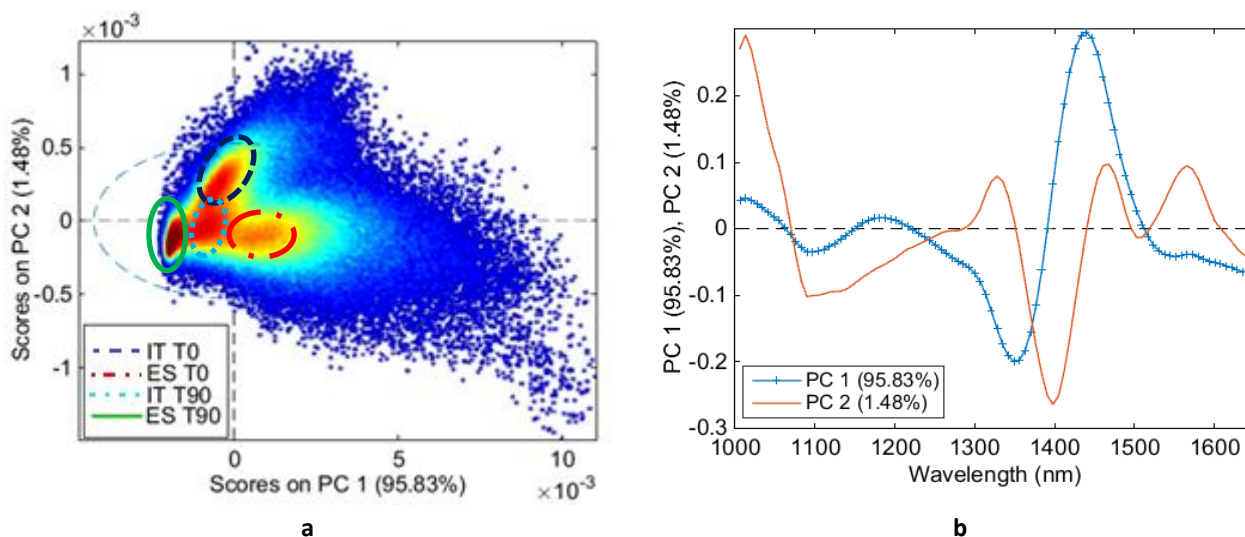


Fig. 5 Score plot of PC1-PC2 (a) and loadings plot (b).

The analysis of PC1 loading plot (Figure 5b) shows the presence of a negative peak around 1350 nm corresponding to CH₃ combinations [26]. The presence of a positive peak around 1400 nm is due to the second overtone of the O-H stretching vibration of H₂O [30]. Analyzing the graph of PC2 loadings, negative peaks, around 1200 nm, are present, they correspond to aromatics (C-H) functional group (second overtone of the functional groups of C-H such as CH₂ and CH₃ bending that are concentrated between 1140-1220 nm for CH₂ and between 1120-1190 nm for CH₃) and the second overtone of the O-H stretching vibration of H₂O around 1400 nm [31-33]. There is also a negative peak around 1100 nm; the range between 1100 nm and 1200 nm corresponds to the second overtone of the functional groups of C-H such as CH₂ and CH₃ bending that are concentrated between 1140-1220 nm for CH₂ and between 1120-1190 nm for CH₃ [31-33]. The presence of a positive peak around 1500 nm can be also detected. Such a behavior is due to the third overtone region of N-H stretching vibration [31,34].

The simple correlation coefficients for pH were consistent with the absorption band related to water and OH absorption band (around 1400 nm) [26,31].

3.3 Partial Least Squares (PLS) model results

The results of the NIR-spectra-collection-based models and the resulting predictions for the chemical and physical parameters are presented in Table 2. Calibration plots of predicted and measured values are shown in Figure 6.

Good correlations were obtained for all the investigated parameters, with r^2 values of 0.93, 0.89 and 0.96 for pH, STOC and STN, respectively. The RMSEC and RMSECV were also acceptable for the parameters, confirming that the calibration models have a high degree of accuracy and in particular for STN.

Table 2 – **Partial Least Squares (PLS)** summary results related to the measured parameters.

Parameter	Pre-Processing	Cross Validation	No. of samples	Number of Latent Variables (LV)	r^2	RMSEC	RMSECV
pH	Smoothing+2 nd Derivative+MC	Venetian blinds	36	4	0.93	0.14	0.16
Soluble Total Organic Carbon	Smoothing+2 nd Derivative+MC	Venetian blinds	36	3	0.89	0.19	0.22
Total Soluble Nitrogen	Smoothing+2 nd Derivative+MC	Venetian blinds	36	3	0.96	0.05	0.05

RMSEC: **Root Mean Square Error of Calibration** - RMSECV: **Root Mean Square Error of Cross-Validation**
MC: **Mean Centering**

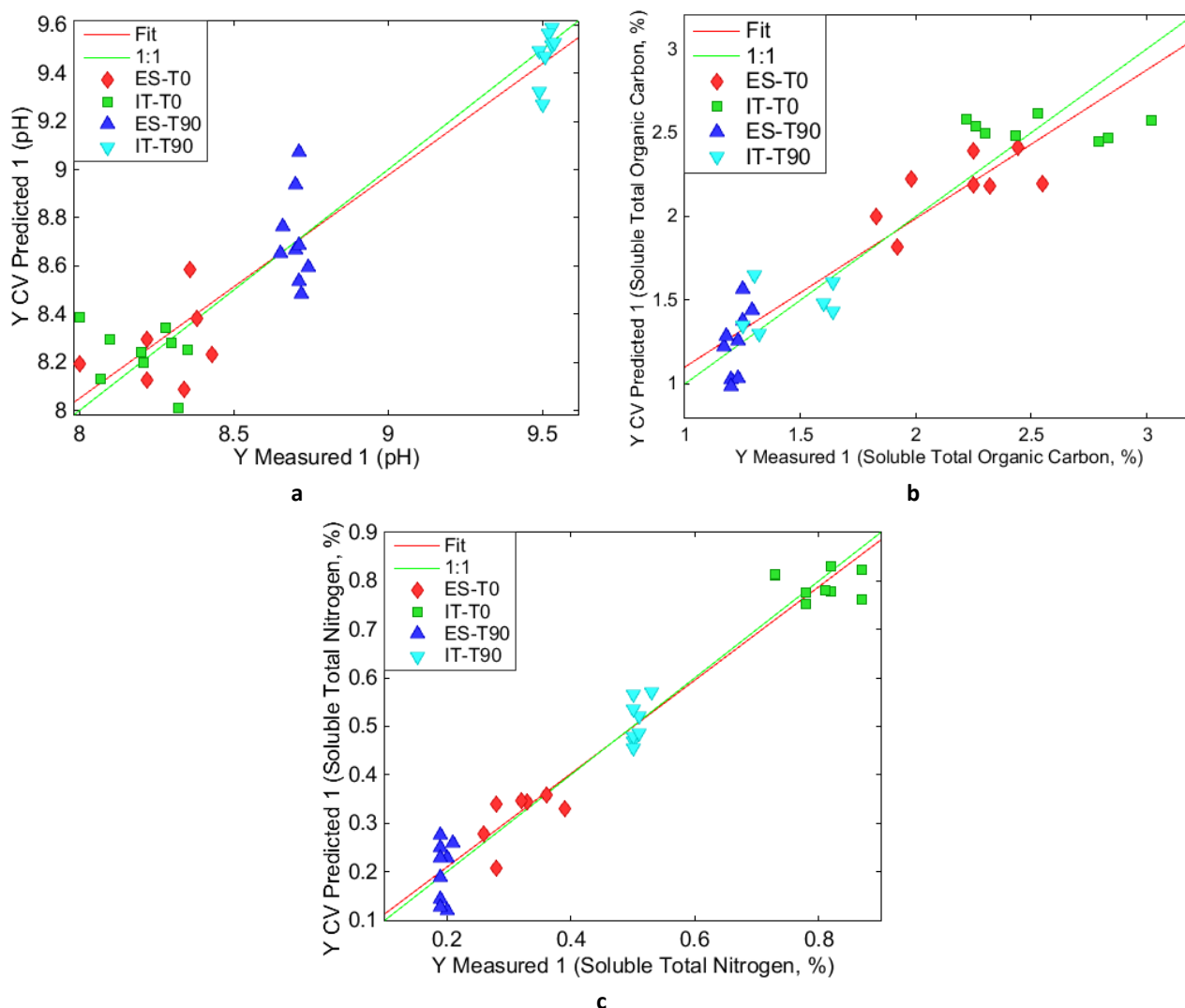


Fig. 6 Scatter plots and regression lines for the **Partial Least Squares (PLS)** models. a: pH, b: Soluble Total Organic Carbon (STOC) (%) and c: Soluble Total Nitrogen (STN) (%).

4. CONCLUSIONS

The study was carried out to investigate the combined utilization of chemical analysis and chemical imaging based techniques to perform a characterization of a new fertilizer. To reach this goal biowaste-derived fertilizer samples were collected at the beginning (T=0) and at the end (T=90) of the curing process. pH, Soluble Total Organic Carbon (STOC) and Soluble Total Nitrogen (STN) were investigated in order to find correlations with the acquired spectral signatures in the NIR wavelength range (1000-1700 nm). The results showed as the proposed **HyperSpectral Imaging (HSI)** based technique, working in the NIR region (1000-1700 nm), combined with a chemiometric approach specifically addressed to perform a spectral based **Partial Least Squares (PLS)** modelling can be very powerful to define and to quantify soil characteristics. Good correlations were found between collected samples and classical laboratory measured parameters. This result is very promising in order to improve analytical speed, specifically during the monitoring of fertilizers evolution (i.e. achieved maturity).

Further studies will be addressed to improve the precision of calibration models through the acquisition and analyses of a larger number of.

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