

Determination of Organic Matter Mineralization in Compost Amended Soils Using FT-NIR Spectroscopy

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ABSTRACT

This study is aimed at developing a non-destructive method for determining organic matter mineralization using FT-NIR (Fourier Transform-Near Infrared) spectroscopy. Soils with two different textures were mixed with olive solid waste (OSW), OSW composts and nitrogen, phosphorus and potassium (NPK) fertilizer. Samples have been incubated at 25 °C for 9 months. After this period, some chemical and physical analyses were performed on the samples using standard methods. Also reflectance spectra of the same samples were acquired right after the standard measurements using FT-NIR spectroscopy. Calibration models between the standard measurements and the spectral measurements performed on samples were established applying Partial Least Squares (PLS) method. Promising N prediction was obtained for the compost added soil group ($R^2=0.62$ and $RMSECV=0.05$) and good N prediction was performed for the control soil group (0.93 and $RMSECV=0.01$). C was predicted successfully for almost all of the data groups. Prediction of C for the data group that included all the soil groups was performed with a coefficient of determination of 0.88 ($RMSECV=0.27$) in validation. C was predicted with the coefficient of determination of 0.93 ($RMSECV=0.23$) for the compost added soil group. It was shown with this study that NIR spectroscopy has the potential of sensing soil contents non-destructively.

Keywords: FT-NIR spectroscopy, organic matter mineralization, olive solid waste.

INTRODUCTION

Olive oil solid waste (OSW) contains olive pulp, stones, residual oil and vegetative waters. OSW can be a good and available source for soil organic matter in Mediterranean Countries. Approximately, 50-60% of olive by volume is solid waste. In general, seed part of olive has been used extensively as a fuel source in some parts of Turkey. Many studies have reported the toxic effects of oil by-products on plants and soil microbial activity, because of their phenolics, fatty acids and mineral salts contents (Martin et al., 2002). Alternative methods of treating the waste are necessary, and composting to produce a soil conditioner is identified as a viable alternative approach.

NIR spectroscopy has good potential to be used as a way of predicting soil quality components non destructively (Bogrekci and Lee, 2007; Maleki et., 2006; Terhoeven-Urselmans et al., 2005; Saeys, 2005).

NIR spectroscopy has advantages for analysis of soil samples in laboratory conditions without using any chemicals and with fast applications resulting multi reading with a single measurement; also, it has the potential to be developed for on line soil sampling needed for site-specific fertilizing applications in the field.

The aim of this research was to evaluate soil organic matter mineralization after the addition of OSW and OSW compost by using FT-NIR spectroscopy.

MATERIALS and METHODS

Soil Compositions: Soil compositions given in Table 1 below were used in this study. Soils had two different textures, Clay Loam and Loam. Treatments were Control soils (clay loam and loam with no organic addition), compost added soils (clay loam and loam) with three different percentages of compost (3, 5 and 7 % respectively), olive soil waste (OSW) added soils (clay loam and loam) with three different percentages of OSW (3, 5 and 7 % respectively), and soils mixed with the combination of OSW and the fertilizer containing nitrogen, phosphorus and potassium with the percentages of 3, 5 and 7 respectively.

Composting Olive Solid Wastes: Three different ratios of OSW were mixed with manure, alfalfa and straw to make compost under controlled conditions. Composts samples (Fig 1) dried at 70 C°, ground and mixed with soil samples before incubation.

Detailed statistics of the N and C contents of soil groups used in the study are given in Table 2.

Table 1. Soil compositions used in the study

	Soil Types										
	Control soil		Compost added*			OSW added*			OSW+NPK added*		
	Clay Loam	Loam	3	5	7	3	5	7	3	5	7
Percentages (%)	100	100	3	5	7	3	5	7	3	5	7
Number of samples	3	3	5	6	6	6	6	6	6	6	6
Total	6		17			18			18		
Total of all	59										

OSW: olive solid waste, NPK: Nitrogen, phosphorus and potassium fertilizer

*Each group having soils from both Clay Loam and Loam



Fig. 1. A prepared compost sample which was used FT-NIR spectral measurements

Table 2. Statistics of parameters of soil groups

Data	Number of samples	Statistics	N (%)	C (%)
Control soils	6	Average	0.196	2.991
		Standard Dev.	0.035	0.908
Compost added soils	17	Average	0.141	3.547
		Standard Dev.	0.083	0.565
OSW added soils	18	Average	0.127	3.540
		Standard Dev.	0.066	0.866
OSW+NPK added soils	18	Average	0.125	3.577
		Standard Dev.	0.055	0.829
All groups in one	59	Average	0.137	3.498
		Standard Dev.	0.067	0.781
Control soils+Compost added soils+OSW and NPK added soils	41	Average	0.142	3.479
		Standard Dev.	0.069	0.752

OSW: olive solid waste, NPK: Nitrogen, phosphorus and potassium fertilizer

Spectral measurements were performed in reflectance mode using a Bruker MPA (Multi-Purpose Analyzer) FT-NIR spectrometer (Fig. 2) (Bruker Optik, GmbH, Ettlingen Germany) equipped with an InGaAs detector and a 20 watts high intensity tungsten-halogen NIR light source. Wavelength region scanned with the fiber optic probe (type IN 261) was from 780 nm to 2500 nm. Thirty two scans were performed per spectrum. Resolution was 8 cm^{-1} . Thirty two scans in about 15.32 s was performed per spectrum. Instrument control and spectra analysis were performed using OPUS software (Bruker Optik, GmbH, Ettlingen Germany). In all spectral measurements, the Blackman-Harris-3-term apodization function, a phase resolution of 64 cm^{-1} , a power spectrum phase correction method and a zero filling factor value of 2, were used.



Fig. 2. FT-NIR spectrometer used in the study

Reflectance spectra were obtained from both reference (Spectralon[®]) and sample consecutively for each sample. Fiber optic probe was placed directly on the equatorial surface of the fruit during spectral measurements. The fiber optic probe used had a bifurcated optical configuration which guided the light to the sample by the source fibers and received the reflected light with the detector (TE-InGaAs) fibers (Fig. 3). In the measuring head of the fiber optic probe, the source and detector fibers were mingled randomly forming a sensing area of about 11.7 mm².

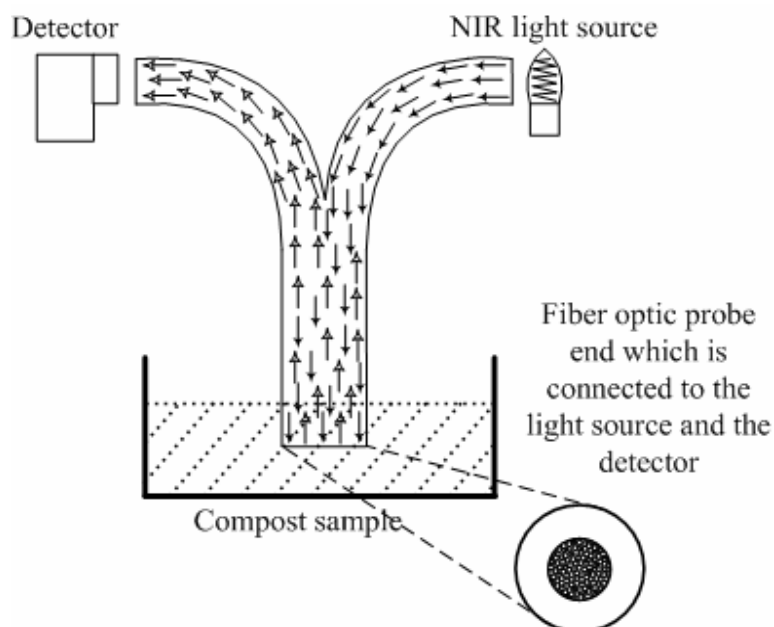


Fig. 3. Schematic view of FT-NIR spectroscopic measurements

Relative spectra of the samples were obtained dividing the sample spectra to the spectra of reference.

The optimal sub wavelength ranges that would yield the best correlations between the physical parameters and the spectroscopic measurements were sought in the wavelength range between 780 and 2500 nm. The partial least squares (PLS) analysis was performed in sub wavelength ranges in the full spectrum of samples. Calibration and validation models were developed using PLS method based on leave-one-out cross validation technique for predicting N and C percents in soil groups.

Different processing techniques were applied on olive spectra in addition to testing different wavelength ranges in PLS analysis, to determine if these techniques would improve the performances of the calibration models. First of all, the relative spectra were used without any pre-processing except smoothing. Second, the following spectrum pre-processing techniques were applied on the olive spectra, one at a time: constant offset elimination, min-max normalization, vector normalization, straight line subtraction, first derivative, second derivative, multiplicative scattering correction. Finally, two processing techniques, one after another, were applied on the olive spectra: first derivative and straight line subtraction, first derivative and vector normalization, and first derivative and multiplicative scattering correction. Mean centering was applied on all kind of spectra processed or not.

Establishing calibration models and performing validations were done using OPUS software. The coefficient of determination, the root mean square error of estimation (RMSEE), and the root mean square error of cross validation (RMSECV) were used to evaluate the performance of the calibration models.

Soil components given in Table 2 were included in the PLS analysis performed. Some statistics of soil data groups are given in Table 2.

RESULTS and DISCUSSION

Good models were obtained in calibration and validation procedures in the result of PLS analyses. Results obtained for each soil composition group and for each quality parameter are given in Table 3.

Although the prediction models developed for N were not as successful as those for C prediction, promising N prediction was obtained for the compost added soil group and good prediction was obtained for the control soil group: coefficients of determination and RMSECV values were as follows for these two soil groups respectively, 0.62 (RMSECV=0.05) and 0.93 (RMSECV=0.01) (Table 3 and Figure 4a-b).

As can be seen from Table 3, C was predicted successfully for almost all of the data groups. Prediction of C for the data group that included all the soil groups was performed with a coefficient of determination of 0.88 (RMSECV=0.27) in validation (Fig. 5a). In calibration on the other hand, R^2 and RMSECV values were 0.93 and 0.21, respectively. C was predicted with the coefficient of determination of 0.93 (RMSECV=0.23) for the compost added soil group (Table 3 and Fig. 5b).

Table 3. Optimal prediction (calibration) results based on FT-NIR spectroscopy

Parameters	Data Groups						
	1	2	3	4	5	6	
Validation	R ²	0.44	0.62	0.55	-	0.93	-
	RMSECV	0.06	0.05	0.04	-	0.01	-
Calibration	R ²	0.52	0.91	0.71	-	0.99	-
	RMSEE	0.05	0.03	0.04	-	0.01	-
# of latent variables	2	6	3	-	8	-	
Data processing	MSC	FDMSC	MMN	-	MSC	-	
Effective wavelength range(s) (nm)	1333-1640, 1835-2175	2173-2355	2260-2355	-	1640-2175	-	
Validation	R ²	0.88	0.88	0.93	0.92	0.92	0.89
	RMSECV	0.27	0.19	0.23	0.24	0.24	0.24
Calibration	R ²	0.93	0.90	0.95	0.99	0.99	0.98
	RMSEE	0.21	0.18	0.20	0.11	0.04	0.12
# of latent variables	6	1	2	8	3	9	
Data processing	MSC	COE	MMN	FDSLS	MSC	NSDP	
Effective wavelength range(s) (nm)	800-1640, 1835-2063	2173-2355	2260-2355	1333-1640, 1333-1470, 1835-1991	1640-2175	800-2175	

1: Includes all the soils compositions, 2: Compost added soil, 3: OSW added soil, 4: OSW and NPK added soil, 5: Control soil, 6: Include control soil, compost and OSW+NPK

OSW: olive solid waste, NPK: Nitrogen, phosphorus and potassium fertilizer. RMSEE: root mean square error of estimation, RMSECV: root mean square error of cross validation. MSC: multiplicative scattering correction, FDMSC: first derivative+multiplicative scattering correction, MMN: min-max normalization, FDSLS: first derivative+straight line subtraction, COE: constant offset elimination, NSDP: no spectral data processing.

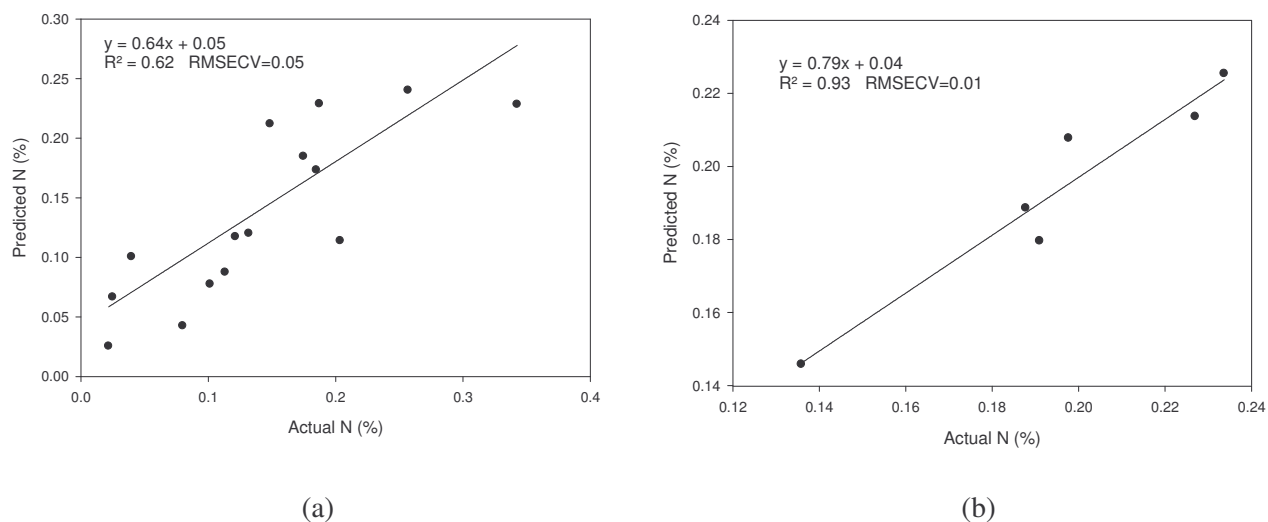


Fig. 4. FT-NIR prediction of N for (a) compost added soil and (b) control soil groups

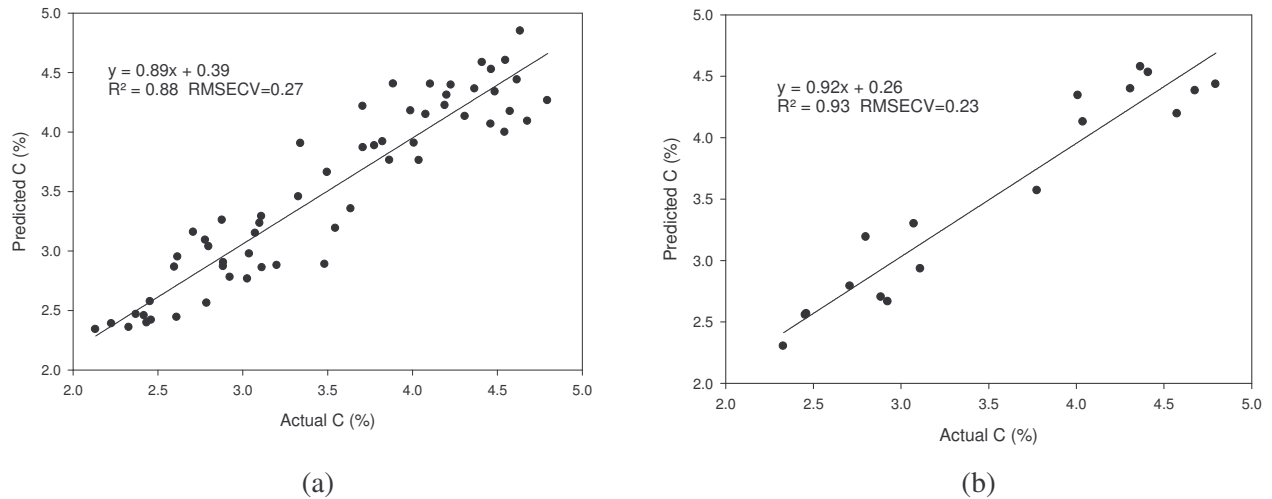


Fig. 5. FT-NIR prediction of C for (a) the soil group, which includes all the soil compositions in it, and (b) compost added soil group

Lower prediction results were obtained for N content of the soil composition groups compared to a previous study (Kavdir, 2008) performed for the prediction of N, C and other quality parameters of OSW composts. However, N prediction was performed successfully for soil groups with no addition.

In most of the cases, RMSECV and RMSEE values given in Table 3 were close to each other, indicating good performance of the model (Lammertyn et al., 1998).

It can be concluded that FT-NIR spectroscopy is a promising technique for non destructive prediction of compost quality. It has a good potential to be applied on line for soil sampling for site-specific fertilizing applications in the field. Therefore, further studies must be performed on this subject.

ACKNOWLEDGEMENTS

The authors acknowledge the financial supports of the Scientific and Technological Research Council of Turkey (TUBITAK, Projects 104O555 and 106O371) for this study.

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