



Change of soil quality based on humic acid with date palm compost incorporation

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Abstract

Purpose The most important factor affecting soil quality is soil humic fraction. Effects of adding carbon in soil humic compounds in arid environments have only been slightly investigated. The change in soil humic acids' structures in relation with manure and palm compost application and the evolution of humic acids' structures during palm composting have not been sufficiently investigated. The purpose of this study is to evaluate if date palm compost is suitable for the sustainable increase of soil carbon content with increasing soil humic compounds.

Methods Soil was amended with date palm compost and sheep manure, which represented two treatments, in addition to a control represented by unamended soil. Soil samples were taken from depth layers of 0–20 cm and 20–40 cm. Physicochemical and spectroscopic analyses were used to study humic acids extracted from organic amendment and soil.

Results The neutral pH and the C/N ratio of 11.9 revealed that the palm/manure compost was mature. Date palm compost application on soil showed its effectiveness in improving soil quality. Spectroscopic studies of humic compounds extracted from date palm compost and manure, showed the abundance of aromatic structures, confirmed by the low E4/E6 ratio associated with an increase in polycondensation and polymerization. Infrared spectroscopy of humic acids extracted from amended soil revealed its enrichment with aromatic structures.

Conclusions Using date palm compost for a short term suggests that this compost may be useful as an alternative to increase the soil fertility by increasing the humic acids quantity with enhancing aromaticity degree and functional groups.

Keywords Humic acids · Soil quality · Compost · Date palm waste · Manure

Introduction

Due to its role in preventing desertification and erosion, organic matter is a basic component of all agro-ecosystems. Organic matter is the result of the combined effect of incorporation, transformation and mineralization of plant and animal residues defined by the local soil and climate characteristics (Horwath 2015). Higher temperature levels accelerate organic matter decomposition; this is likely to cause a serious agriculture problem in the future (Conant et al. 2011).

Several authors showed that soil in Tunisia suffer from low organic matter levels, especially in arid areas (Gargouri et al. 2013). Nevertheless, many organic farms in the Southeast of Tunisia have many problems such as higher soil salinity and low soil fertility (Kouki and Bouhaouach 2009). Among possible solutions, agronomists are looking for amendment of the natural ecosystem to improve the soil quality of agricultural land for crop production.

Among different organic amendments, animal manures offer a readily available solution to soil fertility problems (Eghball 2001). Moreover, a combination of manure with biosolids is considered the best management application for farmers (Rigane and Medhioub 2011).

In the Southeast of Tunisia, date palm is the dominant crop system for many farmers. Agronomists produce organic fertilizers based on date palm waste to protect the environment from palm waste and to enhance soil properties. To define the best management practices for farmers, it was

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necessary to examine soil properties for saving soil, money, time, and protecting the environment (Visser and Sterk 2007). Several authors such as Khiyami et al. (2008) found that the final compost based on date palm and crab wastes could be a good organic amendment. Ali (2008) reported that the addition of date palm compost enhances plant growth and germination. Benabderrahim et al. (2018) evaluated the palm trees' compost influence on alfalfa growth. They determined that this compost improves both of the minerals in alfalfa and soil properties. Therefore, using date palm compost increases soil fertility. Yet, effects of adding carbon in the stable soil humic fractions in arid environments have only been slightly investigated. In fact, the most important factor affecting soil quality is soil humic compounds (Xu et al. 2007). They enhance the life of N and P in soil, improve soil aggregates and stimulate plant respiration in soil (Hernandez et al. 2001).

To the best of our knowledge, no work has investigated if date palm compost mixed with animal manure increases soil humic acid compounds for a short term in arid soils. In fact, change in the humic acids' structures in relation with manure and palm compost application and the evolution of humic acids' structures during palm composting have not been sufficiently investigated.

In this paper, physical and chemical properties and structures of humic acids from manure, date palm compost (34% date palm waste and 66% manure) and from soil were used to evaluate compost maturity and soil quality. Humic compounds' evolution in soil and amendments were studied using UV–visible and infrared spectroscopy. Thus, this study aimed to evaluate soil quality after short-term incorporation of palm composts. On the other hand, this work aimed to evaluate if date palm compost is suitable for a sustainable increase of soil carbon content.

Materials and methods

Study area and sampling

The field study was done in part of an organic farm that had a composting unit located in the oasis of Chenini, along the coast of the Gulf of Gabes (33° 53' N, 10° 03' E) in the Southeast of Tunisia. The climate of the region is a Mediterranean arid climate.

Three soil treatments were installed. The first plot was amended with 10 kg of palm compost, while the second received 10 kg of manure and the last one was considered as a control soil without any amendment. The surface of each plot was 1.68 m². The space between plots was about 1.0 m. Soil plots were irrigated every 15 days. Organic amendments were incorporated into the soil and were mixed into a superficial soil layer (0–20 cm).

Samples' collection was made using an auger from two depth horizons 0–20/20–40 cm. All the collected samples were air dried and passed through a 2 mm sieve prior to analytical work.

The compost used was produced in the integrated organic farm.

The raw materials of the compost were oasis palm wastes (34%) and sheep manure (66%). Composting was in wind-row during 6 months until full maturity. Immature compost obtained after 3 months of composting process and mature one obtained after 6 months of composting were used. The manure used for the positive control was a sheep-goat type. For physical and chemical analyses, the amendments were air dried and crushed.

Physical and chemical analyses

The initial mixture, final compost and manure were analyzed. Each value determined the mean of three replications. pH and EC were measured in an amendment–water mixture with a ratio (1:5 w/v) using a pH meter and conductivity meter (AME type 123) at 25 °C, respectively.

Dry matter (DM) content was measured by drying samples at 105 °C until weight constancy; organic matter (OM) was determined by loss of weight on ignition at 400 °C in a muffle furnace providing the total organic matter content. The available phosphorus was measured calorimetrically (Pauwels et al. 1992). Total nitrogen analysis was performed using the Kjeldahl method. Na, Ca and K content were determined first by extraction through heating amendment (1.0 g) with HNO₃ (0.1 N); then, the extract was analyzed by atomic absorption spectrophotometer (Pauwels et al. 1992).

Soil analyses were carried out before organic amendment application in soil and 3 months after. The Robinson pipette method and texture diagram (Jemagne 1967) were used to find particle size distribution and texture, respectively. Organic matter concentration was determined by carbon content and a conversion was done using the multiplication factor of 1.72. The Dichromate oxidation method (Walkley and Black method) was used for the determination of soil carbon concentration (Pauwels et al. 1992).

The extraction of humic compound from composts and manure was carried out using Tomati et al.'s (2001) method. Compost (2 g) was mixed with NaOH solution (200 ml) and stirred. Humic fractions were obtained using centrifugation. The extract containing humic compounds was acidified by HCl. After centrifugation, two fractions were obtained: FA (supernatant) and HA (precipitate). After 24 h, the precipitated HA was dissolved in a NaOH solution at pH 12 and was filtered to find a clear HA solution.

Soil humic acids were determined using the Rivero method (1998). Soil was stirred with NaOH and Na₄P₂O₇ solutions. The HA was separated from the supernatant (FA)

using centrifugation. The decreasing pH was achieved by adding HCl to the solution. The precipitated HA was separated by centrifugation, and then dissolved in NaOH and precipitated by adding HCl solution.

After extraction of HA and FA from different samples, the ratio of HA and FA fractions (C_{HA}/C_{FA}) was calculated.

The obtained solution of different fractions was dialyzed to eliminate excess salt and finally lyophilized to perform a spectroscopic analysis.

Spectroscopic analysis

A mixture of 1 mg humic acid with 400 mg potassium bromide (KBr) was used for infrared spectroscopy analysis. Spectra were determined for a wavelength between 4000 and 400 cm^{-1} .

The E4/E6 ratio was determined by dissolving 3.0 mg HA in 0.05 M NaHCO_3 , adjusting the pH at 8.3 and by measuring at the absorbance at 465 and 665 nm by PerkinElmer model Lambda 15 UV–Vis spectrophotometer.

Statistical analysis

Mean comparison analyses using the ANOVA method were determined using SPSS 13.0 for windows (SPSS Inc.) Analysis of variance was calculated to determine changes in soil and amendment parameters using Duncan's multiple range tests at 5% level of significance ($P < 0.05$).

Results and discussion

Palm compost and manure characterization

The physical and chemical parameters of initial compost, mature compost and manure are determined (Table 1).

These parameters are useful for assessing compost maturity and stability (Raj and Antil 2011). Compost pH decreased during the composting process from 8.33 to 7.16. A neutral pH indicates that compost is mature and stable (Gobat et al. 1998). This decrease can be explained by the release of organic acids with organic matter decomposition and/or CO_2 solubilization (Ultra et al. 2005).

Compost characterized by C/N ratio decreased from 29.12 to 11.40 at the last step of composting process. The value of C/N ratio among 10 and 15 is assumed to be related to compost maturity (Aparna et al. 2008). Therefore, the date palm compost used was a mature one. Under the composting process, the C_{HA}/C_{FA} ratio enhanced from 0.36 to 1.2. However, the C_{HA}/C_{FA} ratio less than 1 indicated that the compost is not mature, which is contradictory to basic assessment using C/N and pH indicators. The C_{HA}/C_{FA} ratio was used for measuring humification level and for evaluating

Table 1 Physical and chemical characteristics of compost and manure

	Initial mixture	Final compost	Manure
Moisture (%)	64.15 ± 0.01^a	38.88 ± 0.03^b	14.80 ± 0.62^c
DM (%)	35.85 ± 0.00^c	61.23 ± 0.23^b	85.2 ± 0.60^a
EC (mS/cm)	7.30 ± 0.21^a	6.55 ± 0.43^b	7.13 ± 0.06^a
pH	8.33 ± 0.01^a	7.16 ± 0.03^c	7.81 ± 0.01^b
OM (%)/DM	85.36 ± 0.05^a	47.85 ± 0.04^c	48.20 ± 0.6^b
TOC (%)/DM	49.52 ± 0.02^a	18.59 ± 0.01^c	21.43 ± 0.04^b
TN (%)/DM	1.7 ± 0.10^a	1.63 ± 0.01^a	1.04 ± 0.01^b
C/N	29.12 ± 0.02^a	11.40 ± 0.06^c	20.6 ± 0.16^b
C_{HA}/C_{FA}	0.36 ± 0.02^b	1.2 ± 0.04^a	Not determined
MM (%)	14.63 ± 0.06^c	52.15 ± 0.06^a	51.80 ± 0.20^b
P_2O_5 (mg/kg)	772.77 ± 0.16^c	1086.91 ± 0.09^b	1740.87 ± 0.07^a
K_2O (mg/kg)	$10,845 \pm 0.1^b$	$12,050 \pm 0.1^a$	9640 ± 1^c
Na (mg/kg)	5250 ± 0.1^b	7000 ± 0.1^a	5000 ± 2^c
Ca (mg/kg)	$20,000 \pm 0.1^c$	$38,000 \pm 0.1^a$	$21,000 \pm 1^b$

compost maturity (Ouatmane et al. 2002). Thus, assessment of compost maturity should be linked not only to C/N ratio but also to humification degree. The compost used in this study was mature and the composting process is efficient. In addition, date palm compost had a high value of available potassium (1.2%), and calcium (3.8%), which are considered as important indicators of compost quality. The manure contained a high amount of phosphorus (0.17%) as well as calcium (2.1%), sodium (0.96%) and nitrogen (1.04%). The mature compost was of a good quality and met the German nutrient quality standards with the following nutrient contents: potassium < 2000 mg/l, avail-N < 300 mg/l and phosphate < 1200 mg/l. These contents fall within the typical range for K between 0.6 and 1.7% and Ca between 1.0 and 4.0% (Lakhdar et al. 2009).

Changes in soil properties after composting application

Particle size distribution determined that both amended and control soils had low clay content of about 2.68%, followed by silt content that was about 17.85%, and the highest sandy content that was about 79.35%. The experimental field had a sandy loam texture. Soil pH was not affected by manure or compost addition (Fig. 1). Soil depth did not affect the soil pH. Results showed that the soil pH values remained above 7 for two amended soils. Organic amendments did not affect soil pH in both of the studied depths of the arid sandy soil that remained alkaline. Indeed, converse impacts were reported by several authors indicating that the incorporation of compost and manure for a long term increases (García-Gil et al. 2004) or decreases (Bastida et al. 2008) soils' pH, according to their initial pH and organic wastes. The buffering capacity could be linked to the high humic content of



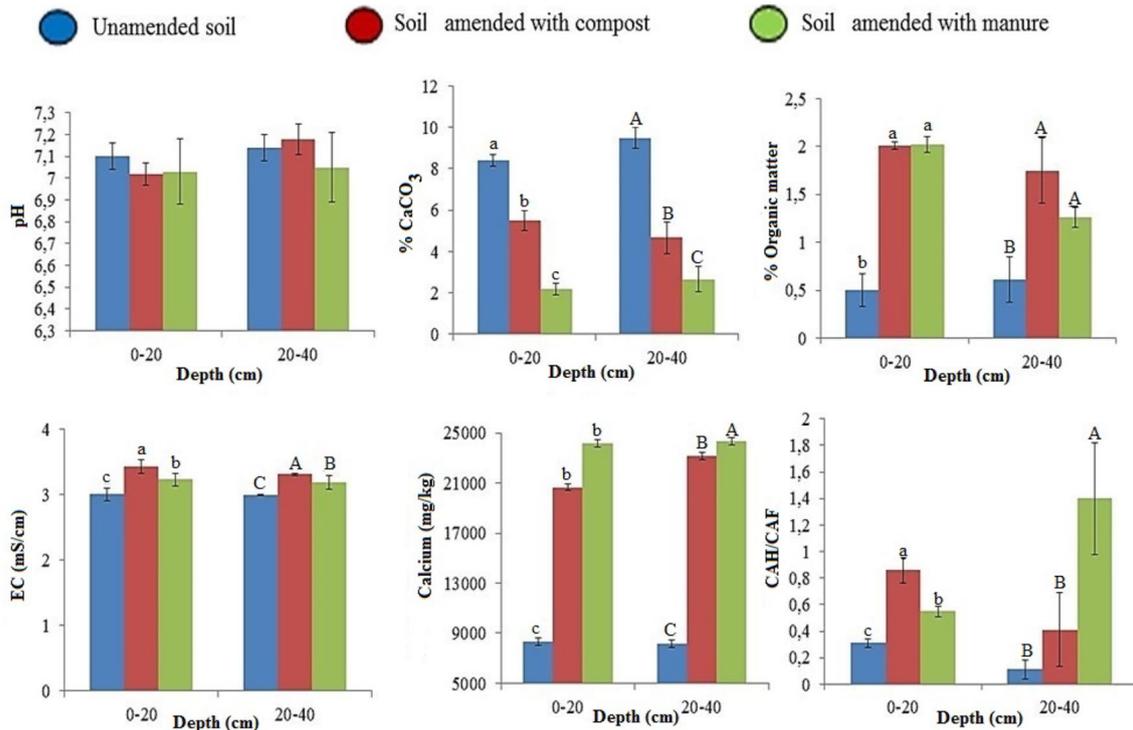


Fig. 1 Variation of soil properties with regard to treatment and depth. Lowercase letters indicate significance within 0–20 cm depth and capital letters indicate significance within 20–40 cm depth. Means with different letters indicate a significant difference at $P=0.05$

compost (García-Gil et al. 2004). Dikinya and Mufwanzala (2010) reported that soil pH did not change with the addition of irrespective dose of chicken manure.

The EC of the control soil was about 3 mS/cm (Fig. 1) with a slight increase in amended soil. This increase was remarkable in the soil amended by compost more than soil amended with manure in both the horizons (0–20 cm and 20–40 cm), although there was no major difference in EC values (6.55 mS/cm for compost and 7.13 mS/cm for manure). The EC increase may be due to organic matter mineralization and transformation. It was reported that EC increases in soil after organic amendments incorporation such as compost and manure due to the addition of soluble salts and organic matter mineralization (Franco-Otero et al. 2011). The increase of EC was found in acidic as well as alkaline soils with the incorporation organic amendment of different natures (Sarwar et al. 2003).

The studied soil had a calcium carbonate content (Fig. 1) of almost 8% at the surface layer of 0–20 cm and 9% at the layer of 20–40 cm. The application of organic amendments to the soil decreased calcium carbonate value. The amounts of CaCO₃ in soil were 5% and 2% for compost and manure, respectively. This reduction was more in soil amended with manure than soil amended by compost. The decrease in CaCO₃ values can be explained by the buffer soil response to acidification results to organic acids. Hemmat et al. (2010)

also indicated the same phenomenon and observed a dose-dependent effect. The CaCO₃ dissolution was also determined by seasonal patterns in Mediterranean environments (Serrano-Ortiz et al. 2010).

The CaCO₃ amended soil had a calcium content about 20.7 (g/kg) and 24.2 (g/kg) for compost and manure, respectively. The addition of organic amendment in soil might help increase calcium content in both the soil layers (0–20 cm and 20–40 cm). In our situation, the increase of Ca²⁺ can be explained by soil CaCO₃ dissolution (Fig. 1) becoming another source of Ca²⁺. Soil organic matter was the most responsible for soils' adsorbing power resulting in an improvement in the cation exchange capacity (CEC) and enhancing granulation. The soil organic matter decomposition process also contributes cations such as Ca²⁺ and K⁺ (Brady and Weil 2005).

The organic matter analysis for both depths showed that a low amount (0.5%) was recorded in control soil, while soil organic matter reached 2% in amended soil for depth 0–20 cm and a value of 1.25% for depth 20–40 cm. The amendment improved the organic matter concentration in soil surface layers with a slight migration in the deep layers. Soil organic matter concentration increased with compost incorporation in soils. The amendment addition clearly increases the level of SOM, which is in agreement with other authors (Heras et al. 2005). The SOM increased at the surface layer (0–20 cm)



and showed a slight increase in the subsurface soil layer (20–40 cm). This can be explained by the addition of organic carbon and its migration into the soil. Soil chemical properties were improved by soil organic matter including nutrients' supply by mineralization and their availability to plants, cation exchange capacity and binding of heavy metals and pesticides (Stangel 1991).

Soil organic matter quality may be evaluated by the C_{HA}/C_{FA} ratio. Humic compounds can be indicative of aggradative or degradative development in soil (Valarini et al. 2003).

Results showed a clear increase of C_{HA}/C_{FA} ratio after incorporation of palm compost and manure. This increase was greater with compost (0.86%) in the surface soil layer (0–20 cm) and conversely with manure (0.55%) in the deeper one (20–40 cm). The increase of ratio C_{HA}/C_{FA} after compost incorporation can be an indication of enhancing carbon content associated with humic acid compounds (Rivero et al. 2004). Palm compost increased humic fractions in soil with increasing C_{AH}/C_{AF} .

Results showed that the compost was more efficient in boosting the humification process than manure. As regards the important role of humic compounds in the soil, compost could be considered as a better fertilizer than manure.

Characterization of humic substances

Date palm compost and manure

Humic acids were extracted from initially processed substrates, produced by palm compost and manure. E_4/E_6 ratios were used as indices to determine humic acids' molecular size. The value of this ratio was inversely proportional to the molecular size and the values are generally five times lower for humic compounds (Amir et al. 2003; Sellami et al. 2008).

UV spectroscopy analysis of humic acids (Table 2) showed a high E_4/E_6 value of about 3.50 in the initial mixture. The E_4/E_6 ratio decreased to 2.27 in the final compost. The ratio was lower in the case of manure (2.08). Spectroscopic E_4/E_6 ratio parameters can be used to assess compost maturity and develop humic acids (Haddad et al. 2015). Tchegueni et al. (2013) reported that E_4/E_6 was 3.96, at the beginning of the composting process and after 3 months, it stabilized at approximately 2.68. The lower value in manure (2.08) indicated greater stability. Lower E_4/E_6 ratios indicate the dominance of aromatic constituents, whereas higher E_4/E_6 ratios reflect the prominence of aliphatic structures (McDonald et al. 2004). Thus, low E_4/E_6 in the case of compost produced indicated

that humic fractions extracted from compost produced have the dominance of aromatic constituents. This can be explicated by the occurrence of an oxidation process characteristic of humification (García et al. 2016).

Data are the mean of three replicates. Values followed by different letters are significantly different according to the Duncan test at $P < 0.05$.

Infrared spectra showed the same spectra at the beginning and the end of the composting process, indicating a conservation of the basic chemical structures during the composting treatment. Figure 2b showed peaks' appearance between 3702 cm^{-1} and 3600 cm^{-1} . Based on Table 3, these signals indicate the presence of two groups: Al–Al–OH free group O–H of phenols ($3650\text{--}3600\text{ cm}^{-1}$) and an aliphatic group. They appeared more intense in the compost-produced spectra.

The two substrates presented a signal around 1700 cm^{-1} which was more intense in the compost spectra. Date palm compost produced presented amines around (1590 cm^{-1}) and aromatic structures around (1510 cm^{-1}), whereas the initial mixture presented secondary amides around (1540 cm^{-1}).

IR spectra of humic fractions for both initial mixture and compost produced (Fig. 2a) showed a reduction in the structures to 2925 cm^{-1} and 2840 cm^{-1} , confirming a microbial use of the aliphatic and peptide structures. However, an increase in the etherified and phenolic aromatic structures was confirmed by an increase in the signal around 1642 and 1371 cm^{-1} .

The HA manure spectra presented two groups located in the area around 1700 cm^{-1} and 1532 cm^{-1} . These signals were relatively more intense in manure than in compost (Fig. 2b). Aromatic structures are developed in the manure, which presented symmetrical and asymmetrical successive bands that were more intense in areas 1673 and 1448 cm^{-1} . For both of the initial mixture and mature compost, infrared spectra were similar and were different just at signals' intensity. This indicates that these materials have similar structures but different functional groups' concentrations.

The signal around 1700 cm^{-1} is mainly due to the vibrations of double $C=C$ in the aromatic structures combined with the carboxylic groupings or carbonyls (Benny et al. 1996). The structural changes indicate biodegradable structures' degradation like polysaccharides, alcohols and acids. This degradation increased aromatic compounds and peptide signals in the palm compost due to micro-organisms activity (Amir et al. 2004). Mature date palm compost can be used as a good amendment.

Soil humic compounds

Infrared analyses for soil humic compounds (Fig. 3) showed a signal around 3700 cm^{-1} . It was the OH–AL–AL group. A relative intensity increase at 3400 cm^{-1} is due to stretching

Table 2 E_4/E_6 ratio of compost and manure HA

	Initial mixture	Produced compost	Manure
E_4/E_6	3.50 ± 0.03^a	2.27 ± 0.02^b	2.08 ± 0.02^c



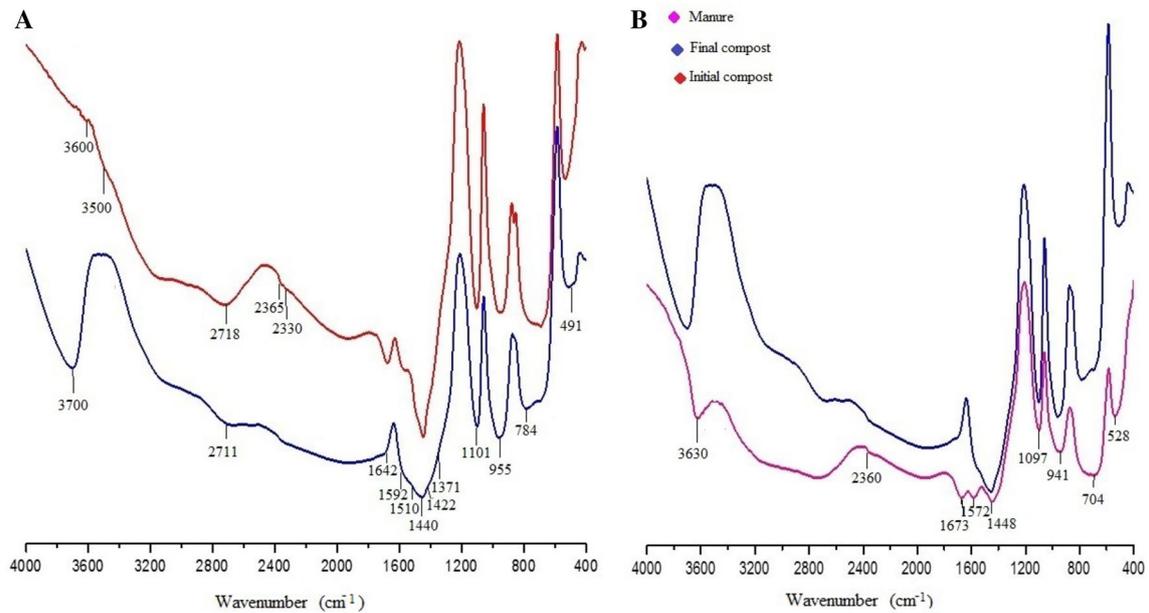


Fig. 2 a Infrared spectra of humic acids extracted from initial mixture and final compost. b Infrared spectra of humic acids extracted from final compost and manure

Table 3 IR bands of major functional groups in humic substances (D’Orazio and Senesi 2009; Abdulla et al. 2010)

Wavenumbers (cm ⁻¹)	Vibration	Groups
3400	O–H	Bound and unbound alcohols (AL); water
3180–3090	NH ₂	I Amine
3080–3030	C–H	CH ₂ aromatic
2920–2850	C–H	CH ₂ et CH ₃ aliphatic
1740–1720	C=O	Aldehydes, ketones, carboxylic acids, esters
1640	C=O	I Amides, carboxylate
	C=C	Alkenes, aromatic
1620–1600	C=C	Aromatic
1600	N–H	Amines
1570–1540	N–H	II Amides
1515–1505	C=C	Aromatic
1425	COO–	Carboxylic acids
1320	C–N	Aromatic I and II amines
1265–1240	C–O	Carboxylic acids, aryl ethers and phenols
	C–N	III Amides
1170–1120	C–OH	Aliphatic alcohols
1080–1010	C–O–C, C–O	Polysaccharides

of vibrations of bound and unbound alcohol and water. Three signals appeared in IR spectra of humic acids under soil mixed with compost characterized by the higher intensity. The signal at 1558 cm⁻¹ was due to stretching vibrations of bands of N–H in secondary amides. The higher intensity at this signal was found in the spectra of humic acid under soil with manure. The signal around 1448 cm⁻¹ represented carboxylic acids grouping. The signal around 1286 cm⁻¹ was stretching C–OH of aromatic groups and it can be due to the

stretching vibrations of ethers and phenols. In spectra of AH extracted from soil amended by manure, signal at 1650 cm⁻¹ indicated the presence of primary amides carboxylic grouping alkenes, and aromatic groups. Besides, the signal at 1540 cm⁻¹ was due to stretching vibrations of bands of N–H secondary amides’ groupings. An increase in the signal at 1065 cm⁻¹ indicated the development of polysaccharides’ grouping. The IR spectroscopic study of humic acids presents amended soil ray spectra having more intense signals



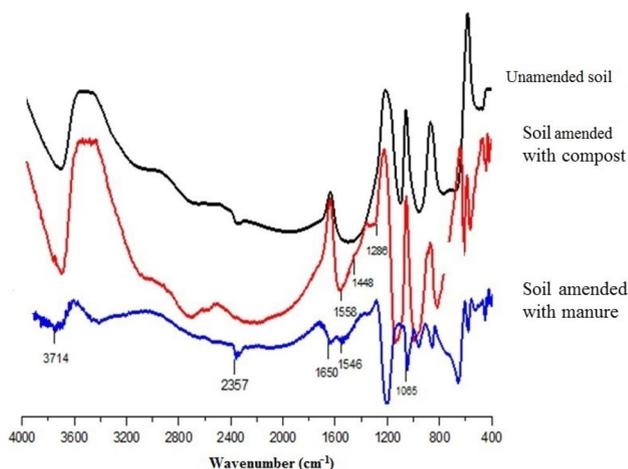


Fig. 3 Infrared spectra of humic acids extracted from unamended and amended soil by manure and compost

in comparison with unamended soil signal including aromatic structures.

These amendments can be used as a source of humic acids' development which largely contributed to soil structure and stability. However, conversely to what was drawn by classical analyses, especially C_{AH}/C_{AF} ratio, manure induced higher aromatic structures' condensation compared to palm compost. Thus, soil amended with manure has more stable humic compounds and therefore a better impact on the soil structure and life. Despite the differences between compost and manure, both of them induced a high humification degree.

Conclusion

The humic components' evolution in the soil in relation with manure and compost application and compost quality are complex and cannot be found by one parameter. Results showed that compost maturity could be evaluated by several parameters: $6 < \text{pH} < 8$, C/N ratio < 15 , C_{HA}/C_{FA} ratio > 1 . IR spectroscopy of humic compounds extracted from compost indicated that aromatic structures increased by the composting process. Thus, improving aromatic structures is an indication of compost maturity. The application of compost to soil showed a slight change in physicochemical properties and fertility levels by higher levels of organic matter, phosphorus and calcium and also by a marked improvement of humification rate. IR spectroscopic analyses applied on amended soils with compost and manure showed a better condensation and polymerization of humic substances. Land application of compost palm waste for a short term provides organic matter nutrients, and improves aromatic structures in sandy soils, especially that sandy soil has high infiltration rates and retains little water. Date palm compost can be

useful as an alternative solution to improve soil quality and to prevent environment pollution from waste accumulation.

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