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Research article

The long term effect of Olive Mill Wastewater (OMW) on organic matter humification in a semi-arid soil

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ABSTRACT

This study investigates the performance of soil and OMW microfloras in OMW organic matter humification in soil. In order to highlight the role of OMW and soil microfloras in the processes of OMW organic matter humification, either OMW or soil was sterilized with autoclaving. The study was carried out in microcosms of 1l containing 500 g of raw or sterilized soil, to which was added 200 ml of raw or sterilized OMW. After 24 months of incubation, the amount of phenolic compounds in the different microcosms was statistically indifferent compared to the control. However, TG-DTA and FTIR analysis of soil humic acids showed that recalcitrant OMW phenolic compounds remain in soil humus in the microcosm: sterilized OMW + raw soil, even after 24 months. Results show that humic acids detected in presence of OMW microflora are loaded with proteins and carbonated compounds and deprived of phenolic compounds and aliphatics with long chain, while humic acids detected in absence of OMW microflora are loaded with phenolic compounds.

1. Introduction

Olive Mill Wastewater (OMW) is blackish liquid effluent of Olive oil processing, made of a mixture of olive vegetable water or olive juice (40–50%) and water used during the processing in order to facilitate separation between oil and the other olive constituents (Bazoti et al., 2006; Khdair et al., 2019). OMW is characterized by a low pH (3.5–5.5), a high load of organic matter (COD of 45–220 g O_2/l) and phenolic compounds (0.5–24 g/l) (Paraskeva and Diamadopoulos, 2006; Khdair et al., 2019). The effluent production is concentrated in Mediterranean countries especially Italy, Spain, Greece, Turkey, Syria, Tunisia and Morocco. The amount of OMW produced per Tone of processed olives varies from 0.3 to 1.1 m^3, depending on the olive processing system (Tsioulpas et al., 2002; Khdair et al., 2019). Estimation of OMW production in the Mediterranean basin was 8.3 million m^3 in 1987 (Nefzaoui, 1987), 10 million m^3 in 1993 (Hamdi, 1993), 10 to 12 million of m^3 in 1996 (Cabrera et al., 1996) and 30 million of m3 in 2003 (McNamara et al., 2008). In Morocco, OMW is either collected in natural evaporation ponds, released into urban sewage or discharged into the environment especially by traditional mills.

OMW discharge is a great environmental issue in many countries of the Mediterranean basin. Several performing techniques of biological and physicochemical treatment of OMW were established either in laboratory or pilot scales (Casa et al., 2003; Eroglu et al., 2006; Hamdi, 1993; Paraskeva and Diamatopoulos, 2006; Tsioulpas et al., 2002). These techniques have as a major goal to eliminate OMW organic matter and to reduce its load of phenolic compounds considered toxic (Casa et al., 2003; Magdich et al., 2012; Obied et al., 2007; Saadi et al., 2007). Some of these processes undergo extraction or production of valuable molecules like Methane (Hamdi and Garia, 1993), Hydrogen (Eroglu et al., 2006), enzymes (Tsioulpas et al., 2002) and polyphenols (Bazoti et al., 2006; Obied et al., 2007; Yangui and Abderrabba, 2018). Yangui and Abderrabba (2018) with the use of green adsorbents obtained an efficiency of extraction of 75% of OMW total phenols and 90.60% of hydroxytyrosol which is the most valuable compound. However, due to the huge amounts of OMW produced during a limited duration (October–March) and due to the cost of treatments, the field application of those techniques is rather restricted.

OMW treatment with the bioreactor soil through a spreading to agricultural lands is an interesting alternative of OMW treatment and valorization since it is low cost compared to the other techniques and

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that it reduces clean water demand and improves soil fertility, due to the addition of organic matter and nutrients (Zema et al., 2019). In nature, ripe olives fall naturally into the soil and get biodegraded in it. OMW is loaded with organic matter and fertilizing elements such as N, P, K and Mg (Ayoub et al., 2014; Cabrera et al., 1996; Casa et al., 2003; Paraskeva and Diamatopoulos, 2006; Zema et al., 2019) and it doesn't contain high loads of heavy metals nor pathogenic microorganisms (Ben Sassi et al., 2006). The soil aggregate stability index after irrigation with OMW is higher probably due to OMW organic matter content (Zema et al., 2019). Studies that tackled OMW spreading to soil have well focused on the impact of spreading on physicochemical properties (Cabrera et al., 1996; Mekki et al., 2007; Sierra et al., 2001; Zema et al., 2019) while less interest was addressed to microbial characteristics (Mekki et al., 2007).

Soil is the most diverse of all ecosystems; billions of bacterial cells (0.2–1.5μm), tens of thousands of protist cells (5–50μm) and kilometres of fungal hyphae typically inhabit a single gram of a typical soil (Geisen et al., 2019). Soil Microflora maintains ecosystem functions and productivity by their contribution to decomposition processes and humic substances synthesis, nutrients cycling and bioremediation of toxic metals or other hazardous wastes (Mekki et al., 2007; El Hassani et al., 2010; Federici et al., 2017; Zema et al., 2019). Multi-cellular soil animals (60 μm–17 mm) are conventionally separated into microfauna (e.g., nematodes), mesofauna (e.g., collembolans) and macrofauna (e.g., earthworms) (Korboulewsky et al., 2015). Plants structure soil physics, chemistry and other soil biota due to litter inputs and root growth (Litalien and Zeeb, 2020). Plants roots exudates are responsible for rich microbial diversity around the root zone and provide nutrition to rhizobacteria and mycorrhizal fungi which in turn promote plant growth through diverse mechanisms, which include transformation and acquisition of resources like nitrogen (N) and phosphorus (P), production of phytohormones and enzymes, and mitigation of plant stresses (Vimal et al., 2017).

Fertilizer inputs may change the relative bioavailability of soil C, N and P (Magid et al., 1996; Mekki et al., 2007; Zema et al., 2019). Microbes possess competitive advantage over plants in the acquisition of nutrients from soil (Magid et al., 1996). The acquisitions of organic C, N and P by soil microbes is mainly determined by the activity of four microbial enzymes: β-D-glucosidase, Leucine aminopeptidase and β-N-acetylglucosaminidase, and phosphatase (alkaline or acid) (Magid et al., 1996; Zheng et al., 2020). Considering a proper proportion of inorganic and organic fractions in fertilizers is important to assure environmental sustainability since adding too much organic fertilizer may lessen microbial growth (Zheng et al., 2020).

The study of the microbial impact of OMW application to soil was limited to determination of CO_2 production and microbial groups abundances (Chatzistathis and Koutsos, 2017; El Hassani et al., 2010; Magdich et al., 2012; Mekki et al., 2007; Saadi et al., 2007; Sierra et al., 2001; Federici et al., 2017). In these studies, no distinction between soil microflora and OMW microflora was settled. After OMW application to soil, the obtained changes in microbial population were most of the time attributed to soil microflora as soil was the dominant phase.

This study tackles the contribution of OMW and soil microbial groups at OMW organic matter humification in soil. Soil organic matter is among the most important factors responsible for soil fertility and land protection from degradation, especially, in arid and semi-arid areas. Humic substances are the most abundant and active reserve of soil organic matter (Francioso et al., 2007; Federici et al., 2017). The humic substances components of organic amendments are characterized by higher aliphatic character and molecular heterogeneity, lower contents of O, acidic functional groups and organic free radicals, and lower degrees of aromatic ring polycondensation, polymerization and humification than native soil humic substances (Senesi et al., 2007). The analysis of soil receiving OMW as an organic amendment has to consider the humic substance fractions contained, which is an important indicator of the maturity and stability achieved by organic matter in soil. In this study, we investigated the long term effect of OMW as an organic amendment on soil humic acids and considered the presence and absence of OMW and soil microfloras and their role in the humification process.

2. Materials and methods

2.1. Origin and characteristics of Olive Mill Wastewater

OMW was purchased from a press semi-modern unit located in Fez-Morocco. The effluent pH was 4.70, its COD was 120 g/l and its load of phenols was 7.33 g/l. OMW was filtered three times through glass wool to avoid preferential orientations in microcosms and was used without any dilution.

2.2. Origin and characteristics of soil

Soil was collected in 0–20 cm deep from a field located in the semi-arid Saïs valley (Morocco) using a soil auger from three different sites then the three samples were mixed. Soil was air dried and sieved through 2 mm before use. The soil was a sandy loam constituted of 51.16 ± 5.88% of sand, 32.56 ± 3.92% of silt and 16.25 ± 1.96% of clay. Its degree of humification was 10.68 ± 2.96 (%). Soil PH was 7.9 and its electrical conductivity was 3.11 ms/cm.

2.3. Microcosms conception and incubation

Microcosms were glass containers of 1 l with closer and without any drain, previously sterilized with autoclaving at 120 °C for 20 min. Soil was distributed into microcosms at a rate of 500 g each. OMW was added to soil at a rate of 400 ml/kg. To study the role of OMW and soil microfloras at the process of OMW organic matter humification in soil, we prepared microcosms where soil or OMW are sterilized. Sterilization of soil or OMW was done with autoclaving at 120 °C for 15 min (Hafidi et al., 2005). The created microcosms are: distilled water + raw soil (control), raw OMW + raw soil, raw OMW + sterilized soil and sterilized OMW + raw soil. Microcosms were incubated at 20 °C in a thermostatically controlled incubator and were periodically homogenized using a sterile spatula under aseptic conditions. Experiments were done in triplicate.

Table 1. Dosage of phenolic compounds (mg/g of dry soil) in microcosms. Symbols *, ** refer to statistical comparison at a probability level of 0.05.

	Control (Distilled water + raw soil)	Raw OMW + raw soil	Raw OMW + sterilized soil	Sterilized OMW + raw soil
1 day	0,07 ± 0,03 *	2,68 ± 0,15 **	_	_
40 days	0,04 ± 0,01 *	0,12 ± 0,01 **	0,13 ± 0,02 **	0,13 ± 0,03 **
6 months	0,09 ± 0,01 *	0,67 ± 0,08 **	0,37 ± 0,03 **	0,52 ± 0,05 **
7 months	0,08 ± 0,02 *	0,64 ± 0,06 **	0,36 ± 0,04 **	0,51 ± 0,02 **
12 months	0,12 ± 0,02 *	0,24 ± 0,05 **	0,21 ± 0,07 *	0,24 ± 0,03 **
24 months	0,13 ± 0,03 *	0,17 ± 0,04 *	0,15 ± 0,04 *	0,19 ± 0,06 *

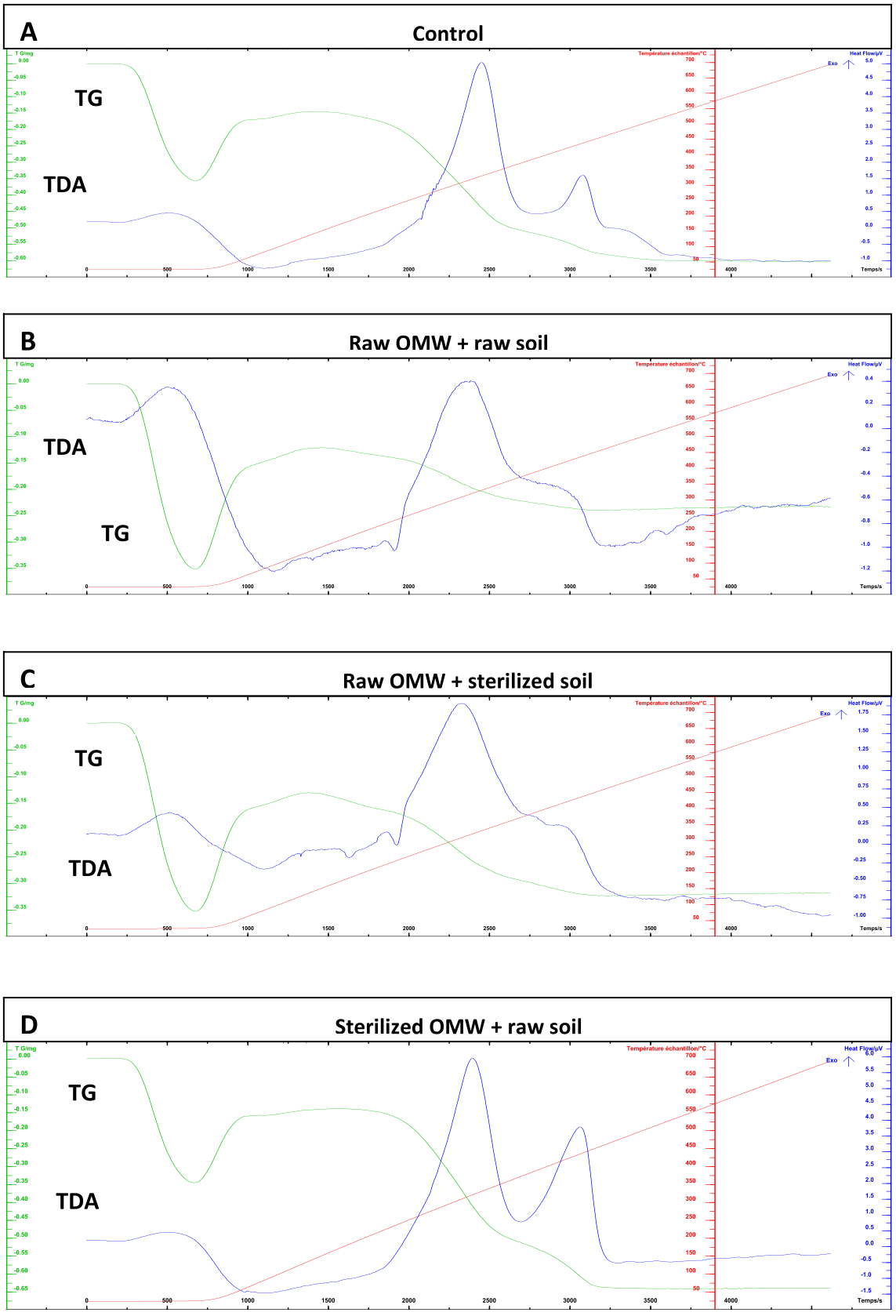

Figure 1. TG-DTA analysis of humic acids of the different microcosms: control (A), raw OMW + raw soil (B), raw OMW + sterilized soil (C), sterilized OMW + raw soil (D).

Table 2. Attributions of absorbance bands in FTIR spectra (Boukir et al., 2001; Hafidi et al., 2005).

Bands and peaks (cm^{-1})	Attribution
3600–3100	Linked OH (ν) (alcohols, phenols, carboxylic acids)
3100–3000	CH (ν) (aromatics)
3000–2800	CH, CH_2, CH_3 (ν) (aliphatics)
1742–1725	C=O (ν) (carboxylic acids, ketones, esters, amides)
1650–1630	C=C (ν) (aromatics)
1400	OH (δ) (phenols), COO^-, CH_3
1200–1100	C–O, OH, C–O–H (γ) (carbohydrates, alcools, aromatics, ethers, Si–O–C groups)
485–1000	C–Cl, C–Br, C–I (halogenures)

2.4. Determination of phenolic compounds in soil

After 40 days, 6 months, 7 months, 12 months and 24 months of incubation, 10 g of soil were sampled from three different levels in each microcosm (up, middle and deep) and used to determine the load of phenolic compounds according to Sierra et al. (2001) by shacking a 1/25 (W/V) mixture of soil/distilled water for 12 h then phenols were determined in the aqueous extract.

2.5. Extraction and characterization of soil humic acids

Soil humic acids were extracted according to Francioso et al. (2007). Humic acids were extracted using Na0H 0.5M then precipitated with H_2SO_4 50%. After dialysis, humic acids were lyophilized.

2.5.1. TG-DTA analysis

Thermal analysis is a continuous and simultaneous determination of mass loss (Thermo-Gravimetric analysis) depending on heat flow (differential thermic analysis). Thermo-Gravimetric analysis (TG) and Differential Thermic Analysis (DTA) of humic acids were accomplished using a BRUKER Vertex 70 apparatus in the CURI center of Fez- Morocco. Thermal analysis was carried out according to Francioso et al., (2005). Alumina crucible was used and samples were isothermally heated to 25 °C for 10 min under airflow (8L/min) then heated from 25 °C to 700 °C in a static air atmosphere at a heating rate of 10°C/min.

2.5.2. FTIR analysis

Infrared (FTIR) spectra are based on the absorption of infrared rays by the analyzed material. Molecules are identified due to the characterization of the vibrations emitted by molecules' bonds. FTIR analysis (MVP2 STAR) was carried out with reflexion attenuated mode (ATR-Diamand) using a BRUKER Advance II 300 in the CURI center of Fez- Morocco. FTIR spectra were obtained at a resolution of 4 cm^{-1} (16 scans).

2.5.3. Statistical analysis

Essays were done in triplicate then the mean and standard deviation were determined. Student test was used to compare means using prism Pad 4 software. The statistical test was performed at $P < 0.05$.

3. Results and discussion

3.1. Determination of phenolic compounds in microcosms

The loads of phenolic compounds in control microcosm, raw OMW + raw soil microcosm, raw OMW + sterilized soil microcosm and sterilized OMW + raw soil microcosm are presented in Table 1. The phenolics level was deeply reduced in different microcosms after 40 days of incubation. This result could be explained by an adsorption that was independent of the sterilization of OMW or soil. In soil, the most likely fraction to adsorb phenolic compounds is organic matter, iron oxides and clay (Jarboui et al., 2008; Yildiz and Gur, 2007). Zema et al. (2019) reported that OMW application may worsen the hydrological response of soil immediately after the spreading. After 12 months of incubation, the load of phenolic compounds in different microcosms stayed significantly higher than control, except for the microcosm raw OMW + sterilized soil, for which the load of phenols was insignificantly different compared to the control. OMW application at 400 ml/kg which is equivalent to a field spreading of 8.80 l/m^2, didn't allow a reestablishment of phenolic compounds load in soil after 12 months. According to this result, an annual spreading of OMW could cause an accumulation of phenolic compounds in sandy loam soils. Federici et al. (2017) reported that after four months from OMW spreading to a Haploxerept soil, phenols amount in soil stayed significantly higher compared to control. Sierra et al. (2007) recommended a limit of 180 m^3/ha of OMW yearly application, because at doses over 360 m^3/ha the phenolic content of soil may increase due to the temporal immobilization of nitrates, which may cause a decrease in plant production. After 24 months of incubation, the amount of phenolic compounds in the different microcosms was statistically indifferent to the control.

3.2. Analysis of humic acids

3.2.1. TG-DTA

The spectra of TG-DTA analyses of humic acids are presented in Figure 1 for the control microcosm (A), raw OMW + raw soil microcosm (B), raw OMW + sterilized soil microcosm (C) and sterilized OMW + raw soil microcosm (D). TG-DTA analysis mentioned three major exothermic peaks (Table 2).

The first peak that appears at 218 °C for the microcosm raw OMW + raw soil and 223 °C for the microcosm raw OMW + sterilized soil must correspond to proteins' denaturation (Mothé; Mothé, 2005). This peak appears in microcosms where OMW microflora exists what could be a consequence of the humus richness with proteins due to the high metabolic activity of OMW microflora. Federici et al. (2017) reported that in soil treated with OMW showed that the fungal abundance was markedly higher than the control soil while bacterial abundances were slightly affected. After organic amendment application, microorganism populations, leading to changes in their quantity and biodiversity, preferentially use the olive mill wastes and all the derived products (Senesi et al., 2007; Federici et al., 2017).

The second exothermic peak that appears at 330 °C for the control would result in decomposition of carbohydrates, proteins and polyunsaturated fatty acids (Dweck and Sampaio, 2004; Francioso et al., 2005, 2007; Gouveia et al., 2004). Mass loss of this peak was low in microcosms where OMW microflora exists.

The third exothermic peak results in decomposition of refractory carbon of aromatic cycles detected through the C=C bonds of phenols (Gouveia et al., 2004; Leinweber and Schulten, 1992). During thermic decomposition, the double bonds of the cycle are broken which leads to the production of saturated structures characterized by a high thermic stability (Francioso et al., 2007). In microcosms where OMW microflora exists (raw OMW + raw soil and raw OMW + sterilized soil), this peak disappeared. This result shows that OMW microflora is performing at biodegrading OMW phenolic compounds and at attacking soil phenolic compounds. In the microcosm sterilized OMW + raw soil, this peak increased in amplitude compared to control, which could explained by an accumulation of recalcitrant and eventually toxic phenols in soil humus. After OMW spreading, the easily-available fraction of water extractable organic carbon was rapidly consumed, whereas the aromatic compounds tended to be accumulated in the amended soils (Federici et al., 2017).

Peaks above 460 °C correspond to the combustion of carbonated residues produced during the previous combustions (Francioso et al., 2007). These residual peaks were more abundant when the organic matter is totally or partially humified by OMW microflora. This result shows that carbonated compounds are abundant in humic acids.

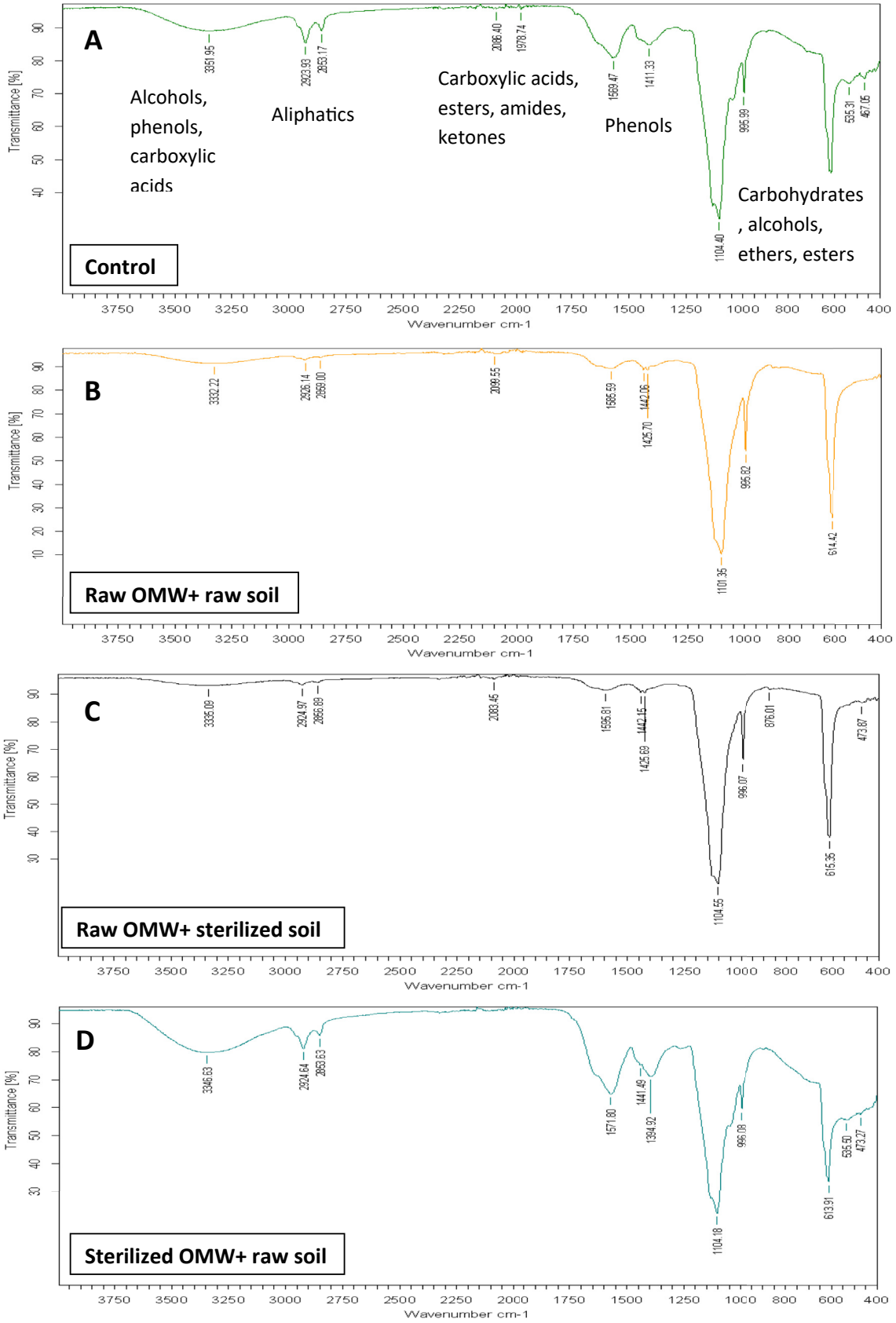

Figure 2. FTIR analysis of humic acids of the different microcosms: control (A), raw OMW + raw soil (B), raw OMW + sterilized soil (C), sterilized OMW + raw soil (D).

Table 3. Major exothermic pics of thermogravimetric analysis (TG) and differential thermic analysis (DTA) of soil humic acids after 24 months of microcosms' incubation.

	1° exo. peak		2° exo. peak		3° exo. peak	
	T_{max} (°C)	Mass loss (%)	T_{max} (°C)	Mass loss (%)	T_{max} (°C)	Mass loss (%)
Control	_	_	330	19,90	438	9,10
Raw OMW + raw soil	218	6,12	319	3,67	_	_
Raw OMW + sterilized soil	223	6,59	309	7,03	_	_
Sterilized OMW + raw soil	_	_	321	23,10	435	8,02

An endothermic peak appeared at 180 °C in the microcosm raw OMW + sterilized soil; it could correspond to an endothermic restructuration of some compounds under the thermic treatment.

3.2.2. FTIR analysis

Spectra of FTIR analysis are presented in Figure 2 for the different microcosms: control microcosm (A), raw OMW + raw soil microcosm (B), raw OMW + sterilized soil microcosm (C) and sterilized OMW + raw soil microcosm (D). FTIR analysis results confirmed those obtained with TG-DTA analysis. Attributions corresponding to FTIR bands and peaks are presented in Table 3.

The band centered between 3351 and 3332 cm^{-1} correspond to the vibration of OH of alcohols, phenols and carboxylic groups (Hafidi et al., 2005; Ricca and Severini, 1993). Phenols appear also in the band centered between 1650 and 1630 cm^{-1} due to the double bonds C=C and around the wavenumber 1400 cm^{-1} due to the vibration of OH of the aromatic. It was noticed that in the presence of OMW microflora, bands corresponding to phenols were deeply reduced. This result shows that humic acids are deprived from phenolic structures because the phenolic compounds of OMW and even those of soil were metabolized by OMW microflora. Phenolic compounds are a favored substrate looked for by the indigenous microflora of OMW. Fungi and bacteria can produce polyphenol-oxidase and β-glucosidase enzymes that have a key role in the degradation of polyphenols and the hydrolytic process during organic matter decomposition (Tsioulpas et al., 2002; Senesi et al., 2007; Federici et al., 2017).

In the microcosm where only soil microflora exists (sterilized OMW + raw soil (Figure 2 D), bands of phenols were ample compared to control (Figure 2 A). This result confirmed the weak performance of soil microflora at biodegrading OMW phenolic compounds which consequently became constituents of soil humic acids.

Bands centered at 2920 cm^{-1} and 2850 cm^{-1} correspond to aliphatic compounds with long-chain detected with CH, CH_2 and CH_3 groups (Francioso et al., 2007). The abundance of aliphatic chains in humus was highly reduced in presence of OMW microflora in the microcosm.

The band centered at 1100 cm^{-1} corresponds to OH groups coupled to C–O and C–OH corresponding to the structure of monosaccharides and polysaccharides (Francioso et al., 2007; Hafidi et al., 2005). OMW application to soil enriches it's humus with carbohydrates mainly due to the metabolic activity of OMW microflora. The second major exothermic peak obtained with TG-DTA analysis reveals that the mass loss was low in the microcosms where OMW exists (Table 3). The low mass loss is likely linked to the nature of carbohydrates (monosaccharides or polysaccharides) in the microcosm.

4. Conclusions

OMW microflora is more performing than soil microflora at biodegrading OMW phenolic compounds. After 24 months of incubation, the amount of phenolic compounds in the microcosms: raw OMW + raw soil, raw OMW + sterilized soil, sterilized OMW + raw soil, was statistically indifferent compared to the control. However, TG-DTA and FTIR analysis of soil humic acids showed that humic acids detected in presence of OMW microflora are loaded with proteins and carbonated compounds and deprived of phenolic compounds and aliphatics with long chain, while humic acids detected in absence of OMW microflora are loaded with phenolic compounds.

Declarations

Author contribution statement

Fatima Zahra El Hassani, Abdelali Fadile, Mouna Faouzi, Abdelah Zinedine, Mohamed Merzouki, Mohamed Benlemlih: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

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Competing interest statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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References

Ayoub, S., Al-Absi, K., Al-Shdiefat, S., Al-Majali, D., Hijazean, D., 2014. Effect of olive mill wastewater land-spreading on soil properties, olive tree performance and oil quality. Sci. Hortic. (Amst.) 175, 160–166.

Bazoti, F.N., Gikas, E., Skaltsounis, A.L., Tsarbopoulos, A., 2006. Development of a liquid chromatography-electrospray ionization tandem mass spectrometry (LC-ESI MS/MS) method for the quantification of bioactive substances present in olive oil mill wastewaters. Anal. Chim. Acta 573, 258–266.

Ben Sassi, A., Boularbah, A., Jaouad, A., Walker, G., Boussaid, A., 2006. A comparison of Olive oil Mill Wastewaters (OMW) from three different processes in Morocco. Process Biochem. 41, 74–78.

Boukir, A., Guiliano, M., Asia, L., Doumenq, P., Mille, G., 2001. Subfractionation, characterization and photooxidation of crude oil resins. Chemosphere 43, 279–286.

Cabrera, F., Lopez, R., Martinez-Bordiu, A., Dupuy de Lome, E., Murillo, J.M., 1996. Land treatment of olive mill waste water. Int. Biodeterior. Biodegrad. 38, 215–225.

Casa, R., D'Annibale, A., Pierruccetti, F., Stazi, S.R., Giovannozzi Sermanni, G., Lo Casio, B., 2003. Reduction of the phenolic components in olive mill waste water by an enzymatic treatment and its impact on durum wheat (*Triticum durum* Defs.) germinability. Chemosphere 50, 959–966.

Chatzistathis, T., Koutsos, T., 2017. Olive mill wastewater as a source of organic matter, water and nutrients for restoration of degraded soils and for crops managed with sustainable systems. Agric. Water Manag. 190, 55–64.

Dweck, J., Sampaio, C., 2004. Analysis of the thermal decomposition of commercial vegetable oils in air by simultaneous TG/DTA. J. Therm. Anal. Calorim. 75, 385–391.

El Hassani, F.Z., Zinedine, A., Mdaghri Alaoui, S., Merzouki, M., Benlemlih, M., 2010. Use of olive mill wastewater as an organic amendment for *Mentha spicata* L. Ind. Crops Prod. 32 (3), 343–348.

Eroglu, E., Eroglu, I., Gunduz, U., Turker, L., Yucel, M., 2006. Biological hydrogen production from Olive Mill Wastewater with two stage processes. Int. J. Hydrogen Energy 31, 1527–1537.

Federici, E., Massaccesi, L., Pezzolla, D., Fidati, L., Montalbani, E., Proietti, P., Nasini, L., Regni, L., Scargetta, S., Gigliotti, G., 2017. Short-term modifications of soil microbial community structure and soluble organic matter chemical composition following amendment with different solid olive mill waste and their derived composts. Appl. Soil Ecol. 119, 234–241.

Francioso, O., Montecchio, D., Gioacchini, P., Ciavatta, C., 2005. Thermal analysis (TG–DTA) and isotopic characterization (13C–15N) of humic acids from different origins. Appl. Geochem. 20, 537–544.

Francioso, O., Ferrai, E., Saladini, M., Montecchio, D., Gioacchini, P., Ciavatta, C., 2007. TG-DTA, DRIFT and NMR characterisation of humic-like fractions from olive wastes and amended soil. J. Hazard Mater. 149, 408–417.

Geisen, S., Briones, M., Gan, H., Behan-Pelletier, V., Frimane, V., Arjen de Groot, G., Hannula, E., Lindo, Z., Philippoth, L., Tiunov, A., Wall, D., 2019. A methodological framework to embrace soil biodiversity. Soil Biol. Biochem. 136, 107536.

Gouveia, D.S.A., Oliveira Santos, J.C., Conceicao, M.M., Dantas Silva, M.C., Prasad, S., 2004. A thermoanalytic and kinetic study of sunflower oil. Braz. J. Chem. Eng. 21, 265–273.

Hafidi, M., Amir, S., Revel, J.C., 2005. Structural characterization of olive mill wastewater after aerobic digestion using elemental analysis, FTIR and 13C NMR. Process Biochem. 40, 2615–2622.

Hamdi, M., 1993. Future prospects and constraints of olive oil mill wastewater use and treatment: a review. Bioprocess Eng. 8, 208–214.

Hamdi, M., Garia, J.L., 1993. Anaerobic digestion of Olive Mill Wastewaters after detoxification by prior culture of *Aspergillus niger*. Process Biochem. 28, 155–159.

Jarboui, R., Sellami, F., Kharroubi, A., Gharsallah, N., Ammar, E., 2008. Olive mill wastewater stabilization in open-air ponds: impact on clay-sandy soil. Bioresour. Technol. 99, 7699–7708.

Khdair, A., Abu-Rumman, G., Khdair, S., 2019. Pollution estimation from olive mills wasteater in Jordan. Heliyon 5, e02386.

Korboulewsky, N., Perez, G., Chauvat, M., 2015. How tree diversity affects soil fauna diversity: a review. Soil Biol. Biochem. 94, 94–106.

Leinweber, P., Schulten, H.R., 1992. Differential thermal analysis, thermogravimetry and in-source pyrolysis-mass spectrometry studies on the formation of soil organic matter. Thermochim. Acta 200, 151–167.

Litalien, A., Zeeb, B., 2020. Curing the earth: a review of anthropogenic soil salinization and plantbased strategies for sustainable mitigation. Sci. Total Environ. 698, 134235.

Magdich, S., Jarboui, R., Ben Rouina, B., Boukhris, M., Ammar, E., 2012. A yearly spraying of olive mill wastewater on agricultural soil over six successive years: impact of different application rates on olive production, phenolic compounds, phytotoxicity and microbial counts. Sci. Total Environ. 430, 209–216.

Magid, J., Tiessen, H., Condron, L.M., 1996. Dynamics of organic phosphorus in soils under natural and agricultural ecosystems. Chapter 11. In: Humic Substances in Terrestrial Ecosystems, pp. 429–466.

McNamara, C., Anastasiou, C., Oflaherty, V., Mitchell, R., 2008. Bioremediation of olive mill wastewater. Int. Biodeterior. Biodegrad. 61 (2), 127–134.

Mekki, A., Dhouib, A., Sayadi, S., 2007. Polyphenols dynamics and phytotoxicity in a soil amended by olive mill wastewaters. J. Environ. Manag. 84, 134–140.

Mothé, M.G., Mothé, C.G., 2005. Characterization of *Agaricus Blazei* by TG/DTA and solid state NMR. Anal. Magn. Reson. 4, 46–50.

Nefzaoui, A., 1987. Contribution à la rentabilité de l'oléiculture par la valorisation optimale des sous-produits, Olivae-IV année-no. 19, pp. 17–28.

Obied, H.K., Bedgood, D.R., Prenzler, P.D., Robards, K., 2007. Bioscreening of Australian olive mill waste extracts: biophenol content, antioxidant, antimicrobial and molluscicidal activities. Food Chem. Toxicol. 45 (7), 1238–1248.

Paraskeva, P., Diamadopoulos, E., 2006. Technologies for olive mill wastewater (OMW) treatment: a review. J. Chem. Technol. Biotechnol. 81, 1475–1485.

Ricca, G., Severini, F., 1993. Structural investigations of humic substances by IR-FT, 13C NMR spectroscopy and comparison with a maleic oligomer of known structure. Geoderma 58, 233–244.

Saadi, I., Laor, Y., Raviv, M., Medina, S., 2007. Land spreading of olive mill wastewater: effects on soil microbial activity and potential phytotoxicity. Chemosphere 66, 75–83.

Sierra, J., Marti, E., Garau, M., Gruanas, R., 2007. Effects of the agronomic use of olive mill wastewater: field experiment. Sci. Total Environ. 378, 90–94.

Senesi, N., Plaza, C., Brunetti, G., Polo, A., 2007. A comparative survey of recent results on humic-like fractions in organic amendment and effects on native soil humic substances. Soil Biol. Biochem. 39, 1244–1262.

Sierra, J., Marti, E., Montserrat, G., Cruanas, R., Garau, M.A., 2001. Characterization and evolution of a soil affected by olive oil mill wastewater disposal. Sci. Total Environ. 279, 207–214.

Tsioulpas, A., Dimoua, D., Iconomoub, D., Aggelis, G., 2002. Phenolic removal in olive oil mill wastewater by strains of Pleurotus spp. in respect to their phenol oxidase (laccase) activity. Bioresour. Technol. 84, 251–257.

Vimal, S.R., Singh, J.S., Arora, N.K., Singh, S., 2017. Soil-plant-microbe interactions in stressed agriculture management: a review. Pedosphere 27 (2), 177–192.

Yangui, A., Abderrabba, M., 2018. Towards a high yield recovery of polyphenols from olive mill wastewater on activated carbon coated with milk proteins: experimental design and antioxidant activity. Food Chem. 262, 102–109.

Yildiz, A., Gur, A., 2007. Adsorption of phenol and chlorophenols on pure and modified sepiolote. J. Serb. Chem. Soc. 72 (5), 467–474.

Zema, D.A., Lucas-Borjab, M.E., Andiloroa, S., Tamburinoa, V., Zimbone, S.M., 2019. Short-term effects of olive mill wastewater application on the hydrological and physico-chemical properties of a loamy soil. Agric. Water Manag. 221, 312–321.

Zheng, L., Hao Chen, H., Wang, Y., Mao, Q., Zheng, M., Su, y., Xiao, K., Wang, K., Li, D., 2020. Responses of soil microbial resource limitation to multiple fertilization strategies. Soil Tillage Res. 196, 104474.