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ORIGINAL ARTICLE

Effects of olive mill wastewater and olive mill pomace on soil physicochemical properties and soil polyphenols

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Abstract

Every year, worldwide olive oil extraction processes produce huge amounts of by-products such as (olive mill wastewater and olive mill pomace) in a short period of time. These products are a major problem that affects the soil. Therefore, the aim of this study was to investigate the single and simultaneous effects of different olive mill wastes and to determine phenolic compounds in soil using (FTIR) spectroscopy. Under laboratory conditions, increasing doses (12.5%, 25%, 50%, 75%, and 100% w/w) of olive mill wastewater (OMWW), olive mill pomace (OMP), and combinations thereof were applied to the soil. A non-significant decrease in soil pH was found, after treatment with olive mill wastewater (OMWW), while a significant decrease was measured after treatment with OMP and the combination of both. Moreover, treatment with OMWW, OMP, and their combination at relatively high doses significantly increased the values of electrical conductivity (EC), organic matter (OM), organic carbon (OC), and phenolic compounds (PP). At low doses, including the legally permissible doses ($50\ m^3\ ha^{-1}\ y^{-1}$ for OMWW and $50–80\ t^{-1}\ ha^{-1}\ y^{-1}$ for OMP), no significant effects were observed on total nitrogen (TN) and assimilable phosphorus (P) in the soil. The FTIR results show that OMWW and OMP have a high content of phenolic compounds. In addition, FTIR analysis provided valuable information on soil components after treatment with OMWW, OMP, and their combination. The overall results show that OMWW and OMP can be considered as helpful amendments and cost-effective fertilizers to improve soil quality.

Keywords Olive mill wastewater · Olive mill pomace · Soil properties · FTIR spectroscopy · Polyphenols

Introduction

The olive tree (*Olea europaea* L.) is one of the most important cultivated crops and represents a significant sector in the economy of the Mediterranean countries, as these countries alone produce about 98% of the world's olive oil [1, 2]. The oil agro-industry produces large quantities of by-products, almost all of which are generated in the Mediterranean basin, with around 30 million cubic meters annually [3]. These by-products are generated yearly in the short time period of 3 or 4 months of the olive harvest [4, 5, 6]. The olive oil production process can be carried out by several methods, including the 3-phase separation process cited in this study, which generates a liquid olive mill wastewater (OMWW) together with a solid olive mill pomace (OMP) [7]. The large quantities of by-products generated by this process cause severe harmful effects to the environment, and management problems in olive oil-producing countries [8, 9, 10].

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The composition of olive mill waste varies greatly and depends on many parameters, such as the ripeness of the olives, the olive varieties, regional characteristics, and the technology of oil extraction (traditional or continuous systems) [11]. Both types of olive mill by-products are characterized by acidic pH, undesirable color and odor, high salinity, high organic load, and high concentration of phenols [12].

Due to these characteristics, the direct disposal of these by-products on the soil must be subject to legal restrictions. The legally recommended dose for land application in most Mediterranean countries is around 50 m^3/ha/year for olive mill wastewater, and 80 m^3/ha/year for olive mill pomace. The disposal of OMW causes serious environmental problems for water, soil, and air, such as the pollution of surface and groundwater due to the high COD value and the presence of polyphenols and heavy metals [13, 14]. Moreover, the application of these by-products directly affects some of the soil properties by increasing soil salinity, phenolic compounds, and electrical conductivity, leading to a decrease in soil pH and fungal communities [15–17].

In addition to the agronomic effects mentioned above, the application of OMWW and pomace to soils could also have important environmental benefits. Previous studies have mentioned that OMWW can be used as a low-cost soil conditioner and fertilizer [18, 19], providing an additional resource of water and nutrients for irrigation and fertilization of Mediterranean olive groves, which are chronically affected by water and organic matter scarcity [14, 18]. As reported by Belaqziz et al. [11] and Vella et al. [20], OMWW can have a positive effect on soil quality, suggesting that it can be used as a natural fertilizer at a concentration of approximately 30 m^3/ha/year or with double dilution.

Furthermore, Lozano-García et al. [21] and Ameziane et al. [22] have demonstrated that the application of olive mill pomace (OMP) can positively affect soil productivity due to its high content of nutrients needed for plant growth and development (i.e., K, N, and P) and organic matter, as well as its high content of macro- and microelements [23]. Literature data on the effects of OMW on soil properties are available, but are not always consistent and in some cases even contradictory [24]. Moreover, there are still gaps in knowledge regarding the influence of OMWW and OMP use on the evolution of phenolic compounds in soil, which have potentially harmful effects on soil physical, chemical and biological properties, the possibility of phytotoxic effects on crops, and contamination of groundwater. Depending on the extraction system, the phenolic compounds contained in olive fruit are lost by almost 53% in OMWW and by almost 45% in OMP [25]

There is a lack of information on the effects of OMWW and OMP applied individually or in combination on soil properties. This information is important because in some Mediterranean countries, they are also applied simultaneously on agricultural land. In addition, the effects of different doses and different treatment durations need to be tested to optimally simulate natural conditions. As it is difficult to fully monitor their use, doses may vary and in certain cases be higher than those permitted by law. The effects of OMWW and OMP on soil properties may also vary depending on the type and properties of the OMWs used, the regional climate and the duration of application.

The agronomic use of OMWW and OMP by direct application to agricultural fields at high doses poses a potential environmental hazard due to their adverse effects on soil properties. However, their potential positive effects on soil productivity must also be mentioned. In this study, we focused on the effects of OMWW and OMP and their combination on soil properties and soil phenolic compounds. For this purpose, we developed a complex experimental design to look into various characteristics of OMW–soil interactions and to help fill some of the knowledge gaps on this topic. The aim of the present study was to investigate the effects of different doses of olive mill wastewater, olive mill pomace and their combination on soil properties. The concentrations tested range from 12.5%, 25% and 50%, which are close to the quantities legally allowed in most Mediterranean countries (50–80 t^{-1} ha^{-1} y^{-1}), to 75% and 100%, which could be reached in real situations, thus covering all possible potential dosages.

Materials and methods

Olive mill waste source and characteristics

The olive mill wastes (OMWW and OMP) used in this study were obtained from the Al Hadja Yamina modern three-phase olive oil mill in the Baghaï region of Khenchela, Algeria, which uses a cold pressing process. The olive mill waste is derived from the Zabouch olive variety and was obtained in November 2019. The main physicochemical characteristics of the olive mill wastewater and olive mill pomace are summarized in Table 1.

Soil source and characteristics

The soil was collected at a depth of 0–20 cm from an untreated apple orchard (35° 29′ 41″ N, 6° 55′ 27″ E) in Khenchela (Algeria). The soil was air-dried, homogenized, and sieved through 2 mm mesh prior to use. The main physico-chemical characteristics of the soil were: pH 7.24 ± 0.02; EC 0.70 ± 0.03 mS cm^{-1}; organic matter 1.92 ± 0.20 g kg^{-1} DM; organic carbon 1.11 ± 0.08 g kg^{-1} DM; total nitrogen 0.17 ± 0.09 g kg^{-1} DM; assimilable

Table 1 Physicochemical characteristics of olive mill wastewater and olive mill pomace

Parameter	Olive mill wastewater Value	Olive mill pomace Value
PH 20 °C	4.78 ± 0.02	4.65 ± 0.02
Electrical conductivity (mS cm^{-1})	9.30 ± 0.01	15.2 ± 0.07
Organic matter (%)	92 ± 0.10	91 ± 0.03
Organic carbon (%)	53.03 ± 0.02	66.13 ± 0.04
Total nitrogen (mg g^{-1})	0.35 ± 0.02	0.7 ± 0.05
Total phenolic contents (mg g^{-1})	87.03 ± 0.1	44.63 ± 0.11

The results are reported as mean ± SD of 3 different measurements

phosphorus 0.04 ± 0.03 g kg -1 DM; phenolic compounds 1.23 ± 0.10 mg kg^{-1} DM; 61% sand, 22% clay, 17% silt.

Experimental design

The experiment was carried out in the laboratory in the growth room under controlled conditions to limit environmental variability. The soil was homogenized and distributed in the containers (PVC cylinders 16 cm × 14 cm × 18 cm) in an amount of 1000 g per each.

Treatment with olive mill wastewater and olive mill pomace and a combination of them

Raw olive mill wastewater was added to the soil in five increasing treatments TW1, TW2, TW3, TW4, and TW5, with treatment TW3 representing the legally allowed dose corresponding to 50 m^3 ha^{-1} $year^{-1}$. Olive mill pomace was air-dried for 48 h before use to reduce moisture content and then also added to the soil in five increasing doses TP6, TP7, TP8, TP9, and TP10. OMP was also tested at the legally recommended dosage (50–80 t^{-1} ha^{-1} $year^{-1}$), which corresponded to treatment TP8. OMP and OMWW were tested in combination with five increasing doses named TWP11, TWP12, TWP13, TWP14, and TWP15. The combination treatments were decided considering the simultaneous application of the different olive mill wastes in the fields, which may occur in several Mediterranean countries. Untreated soil served as control and the experiment was conducted with three replicates per treatment. The microcosms were kept under laboratory conditions for 4 months. The OMWW and OMP and their combinations were mixed with soil in different percentages for the various treatments in the microcosms, as shown in Table 2.

Table 2 The different percentages for the various treatments of OMWW, OMP, and the combination used in microcosms

Treatments	Olive mill waste (%)
OMWW	
TW1	12.5% OMWW + soil w/w
TW2	25% OMWW + soil w/w
TW3	50% OMWW + soil w/w
TW4	75% OMWW + soil w/w
TW5	100% OMWW + soil w/w
OMP	
TP6	12.5% OMP + soil w/w
TP7	25% OMP + soil w/w
TP8	50% OMP + soil w/w
TP9	75% OMP + soil w/w
TP10	100% OMP + soil w/w
Combination	
TWP11	12.5% combination (6.25% of OMWW + 6.25% of OMP) + soil w/w
TWP12	25% combination (12.5% of OMWW + 12.5% of OMP) + soil w/w
TWP13	50% combination ((25% of OMWW + 25% of OMP) + soil w/w
TWP14	75% combination (37.5% of OMWW + 37.5% of OMP) + soil w/w
TWP15	100% combination (100% of OMWW + 100% of OMP) + soil w/w
Control	Untreated soil

Soils physicochemical analyses

At the end of the experiment, the soils in the containers were dried at room temperature, then ground and sieved to 2 mm. They were then analyzed, with all analyses carried out in triplicate.

To characterize the soils, the pH was measured in the aqueous extract using the ratio (1:2.5 soil: water suspension) using a pH meter (EUTECH INSTRUMENTS, Singapore) [26]. Electrical conductivity (EC) was measured in the ratio 1:5 (soil: water suspension) with a multiparameter (HANNA, USA) [27]. Organic carbon (OC) was determined according to the Walkley–Black method by oxidation of the oxidizable matter in the soil with 1 N $K_2Cr_2O_7$. The heat generated when two volumes of H_2SO_4 are combined with one volume of the decrement is assisting the reaction. The remaining dichromate is titrated with ferrous sulfate [28]. The organic matter (OM) was determined by multiplying the total organic carbon by 1.724 [29]. Total nitrogen in soil (TN) was determined by the Kjeldahl method. The measurement of total Kjeldahl nitrogen (N) is carried out in two steps. The first step is the digestion of the sample in concentrated sulfuric acid at high temperatures to convert the mineral nitrogen to the ammonia form, and the second step is the determination of the ammonium in the extract by titration of the NH_3 released by steam distillation [30]. Assimilable phosphorus was analyzed according to the international standard OLSEN NF ISO 11263 [31]. The phenolic compounds were analyzed spectrophotometrically (Agilent Technologies Cary 60 UV–Vis, Malaysia) at 725 nm using the Folin–Ciocalteu method. The soil-soluble phenolic compounds were extracted three times with an equal volume of ethyl acetate. After evaporation of the organic phase, the residue was dissolved in pure methanol and kept at − 20 °C until use. In the soil samples, the phenols were extracted with 80% methanol prior to their purification and analysis [32].

FTIR analysis

FTIR analysis was conducted using a spectrometer (Thermo Scientific Nicolet iS50 spectrometer, France) with the KBr pressed disk technique, which is the most commonly used method. Samples were crushed with dry potassium bromide powder in an agate mortar. The samples were then pressed to form a transparent disk. All spectra were performed from 4000 to 500 cm^{-1}.

Statistical analysis

The measurement of physico-chemical properties of soil after treatment with OMWW, OMP and their combination was carried out in triplicate and the results were expressed as means ± SD. Normality of the data was tested using the Shapiro–Wilk test and homogeneity of variance was assessed using Bartlett's test. As the data were normally distributed, a one-way ANOVA was used to evaluate the effects of the treatments on the soil parameters with XLSTAT 2014.5.03. SAS 9.1 was used to analyze the homogeneous groups using Student's least significant difference (LSD) at 5% significance level. Correlation analysis was carried out to assess the degree of association between the different parameters of the treated soil (pH, EC, OM, OC, TN, P, and PP). The determination of the correlation matrix was defined as a preliminary step to the principal component analysis. The software Statistica 08 was used to perform the principal component analysis (PCA) to analyze the multivariate differences between the treatments and the physicochemical parameters of the soil parameters. Boxplot diagrams were created using Origin Pro software (version 9.0).

Results and discussion

Analysis of variance (ANOVA) (Table 3) revealed that both the separate and combined treatments with OMWW and OMP had a highly significant effect on soil physico-chemical parameters, such as pH, EC, OM, OC, and PP ($p < 0.001$). For P and TN, the effect was not significant.

Effect of OMWW, OMP, and the combination on soil pH

A comparison of the average pH values highlighted the presence of 3 groups, the first (control) being characterized by a neutral pH (Table 4). The evolution of soil pH under treatment with OMWW, OMP, and the combination showed a highly significant decrease with increasing dose (Fig. 1.a).

Table 3 The values of one-way ANOVA from a general linear model analysis of the effects of OMWW and OMP and combination on the soil properties

Source of variation	DF	pH	EC	OM	OC	TN	P	PP
Treatments	15	1.07367556***	0.65104833***	2.10703500***	0.63198611***	0.11205319*	0.73962097*	0.68933278***
Error	32	0.00176042	0.00395833	0.14931250	0.00123750	0.01426042	0.02053333	0.00339167

***Highly significant $p < 0.001$, *Significant, *ns* not significant

Table 4 Physicochemical parameters of soil treated with single and combined OMWW and OMP (mean values of three replications ± SD of each specimen)

Treatments	pH	EC	OM	OC	TN	P	PP
TW1	7.01 ± 0.02c	0.83 ± 0.04a	2.14 ± 0.05a	1.24 ± 0.04a	0.21 ± 0.06a	0.05 ± 0.04b	5.44 ± 0.03c
TW2	6.99 ± 0.01c	0.95 ± 0.02a	2.18 ± 0.08a	1.26 ± 0.08a	0.30 ± 0.06a	0.07 ± 0.02b	5.64 ± 0.04c
TW3	6.76 ± 0.15c	1.11 ± 0.01b	2.47 ± 0.41b	1.34 ± 0.41a	0.46 ± 0.10a	0.09 ± 0.01b	5.89 ± 0.07c
TW4	6.64 ± 0.01b	1.32 ± 0.02b	2.80 ± 0.12b	1.62 ± 0.05a	0.55 ± 0.11a	1.02 ± 0.05a	7.01 ± 0.08e
TW5	6.55 ± 0.02b	1.51 ± 0.03d	3.38 ± 0.03c	1.96 ± 0.05b	0.76 ± 0.20b	1.07 ± 0.02a	9.32 ± 0.04f
TP6	6.66 ± 0.03b	1.26 ± 0.23b	3.54 ± 0.09c	2.05 ± 0.33b	0.28 ± 0.08a	0.06 ± 0.03b	5.74 ± 0.03c
TP7	6.46 ± 0.01b	1.69 ± 0.01d	4.06 ± 0.22d	2.35 ± 0.10b	0.42 ± 0.12a	0.09 ± 0.04b	5.99 ± 0.11d
TP8	6.37 ± 0.02b	1.78 ± 0.02d	4.54 ± 0.06d	2.63 ± 0.10b	0.57 ± 0.04b	0.10 ± 0.02a	5.11 ± 0.10b
TP9	6.35 ± 0.01b	2.00 ± 0.03d	5.09 ± 0.05e	2.95 ± 0.05c	0.65 ± 0.08b	1.07 ± 0.13a	7.41 ± 0.05e
TP10	4.70 ± 0.02a	2.21 ± 0.01c	5.49 ± 0.04e	3.18 ± 0.04c	0.73 ± 0.06b	1.06 ± 0.13a	8.03 ± 0.05e
TWP11	6.73 ± 0.02c	0.94 ± 0.02a	2.97 ± 0.13b	1.70 ± 0.14a	0.23 ± 0.03a	0.05 ± 0.03b	4.69 ± 0.08b
TWP12	6.64 ± 0.03b	1.02 ± 0.03b	3.28 ± 0.10c	1.90 ± 0.07b	0.41 ± 0.08a	0.06 ± 0.03b	4.84 ± 0.05b
TWP13	6.38 ± 0.02b	1.35 ± 0.02b	3.63 ± 0.06c	2.10 ± 0.10b	0.55 ± 0.06a	0.09 ± 0.03b	5.12 ± 0.13b
TWP14	5.94 ± 0.03b	1.70 ± 0.06d	4.48 ± 0.17d	2.95 ± 0.07c	0.62 ± 0.07b	1.01 ± 0.02a	6.58 ± 0.04d
TWP15	5.76 ± 0.01b	2.03 ± 0.01c	5.29 ± 0.06e	3.06 ± 0.05c	0.69 ± 0.13b	1.04 ± 0.01a	6.74 ± 0.09d
Control	7.24 ± 0.02c	0.70 ± 0.03a	1.92 ± 0.20a	1.11 ± 0.08a	0.17 ± 0.09a	0.04 ± 0.03b	1.23 ± 0.10a
LSD	0.0698	0.1046	0.6427	0.0585	0.1986	0.2383	0.0969

With the homogeneous groups

SD standard deviation ($P < 0.05$)

DM dry matter

Units EC (mS cm^{-1}); OM (g kg^{-1} DM); OC (g kg^{-1} DM); TN (g kg^{-1} DM); P: Assimilable phosphorus (g kg^{-1} DM); PP: phenolic compounds (mg kg^{-1} DM)

However, the soil pH is nearly neutral and remained almost unchanged when treated with OMWW. Moreover, a significant decrease in soil pH values was observed in the OMP treatment and the combination treatment compared to the control, including the OMP treatment dose corresponding to 50–80 t^{-1} ha^{-1} $year^{-1}$. The slight change in soil pH due to OMWW treatment, even at the highest dose of 100% OMWW, is probably due to the high buffering capacity of the soil, which neutralized the acidity of the OMWW, for which the presence of organic acids is mainly responsible. These results are confirmed by Meftah et al. [3] who found that the soil pH at 50 cm depth (S50) changed only slightly despite the application of the acidic OMWW (4.46). The carbonate in the soil neutralized the acidic pH of the OMWW by converting to bicarbonate. The acidic effect of OMP on soil pH at a dose of 100% is due to the presence of organic acids (e.g., phenolic acids, fatty acids…) [33, 34]. In contrast to our results, most published studies [22, 35, 36] report only a slight decrease in soil pH after the addition of OMP. The combination treatment also showed a decrease in soil pH. This decrease was mainly due to the acidic nature of both OMWW and OMP, which were present in the combination treatment at different dosages. In some previous studies [37, 38], it was found that soil pH usually decreased after OMWs spraying. This decrease could be explained by the organic acids and polyphenols present in olive mill wastes, as well as on the ripening degree and post-harvest condition of storage [39].

Effect of OMWW, OMP, and the combination on soil EC

A comparison of the average EC indicated the presence of 4 groups, with the control characterized by the lowest soil EC (Table 4). The soil EC significantly increased under all three treatments (OMWW, OMP and the combination), with the highest increase in the 100% OMP treatment and the 100% combination compared to the control. In addition, the OMWW treatment also showed an increase in soil EC. The recommended doses for OMWW and OMP showed a remarkable increase in soil EC (Fig. 1b). In this context, our results clearly confirm previous studies [3, 10, 15] which reported that soil EC increased significantly after OMWW treatment. Generally, the increase of soil EC was mainly attributed to the high salt concentration in the OMWW [39]. Moreover, the effect of OMP on soil EC is dose-dependent, i.e., EC increases with increasing OMP dose. This is mainly due to the high content of soluble salts, particularly potassium salt, in OMP. Our results are in agreement with the results of Ameziane et al. [22], who

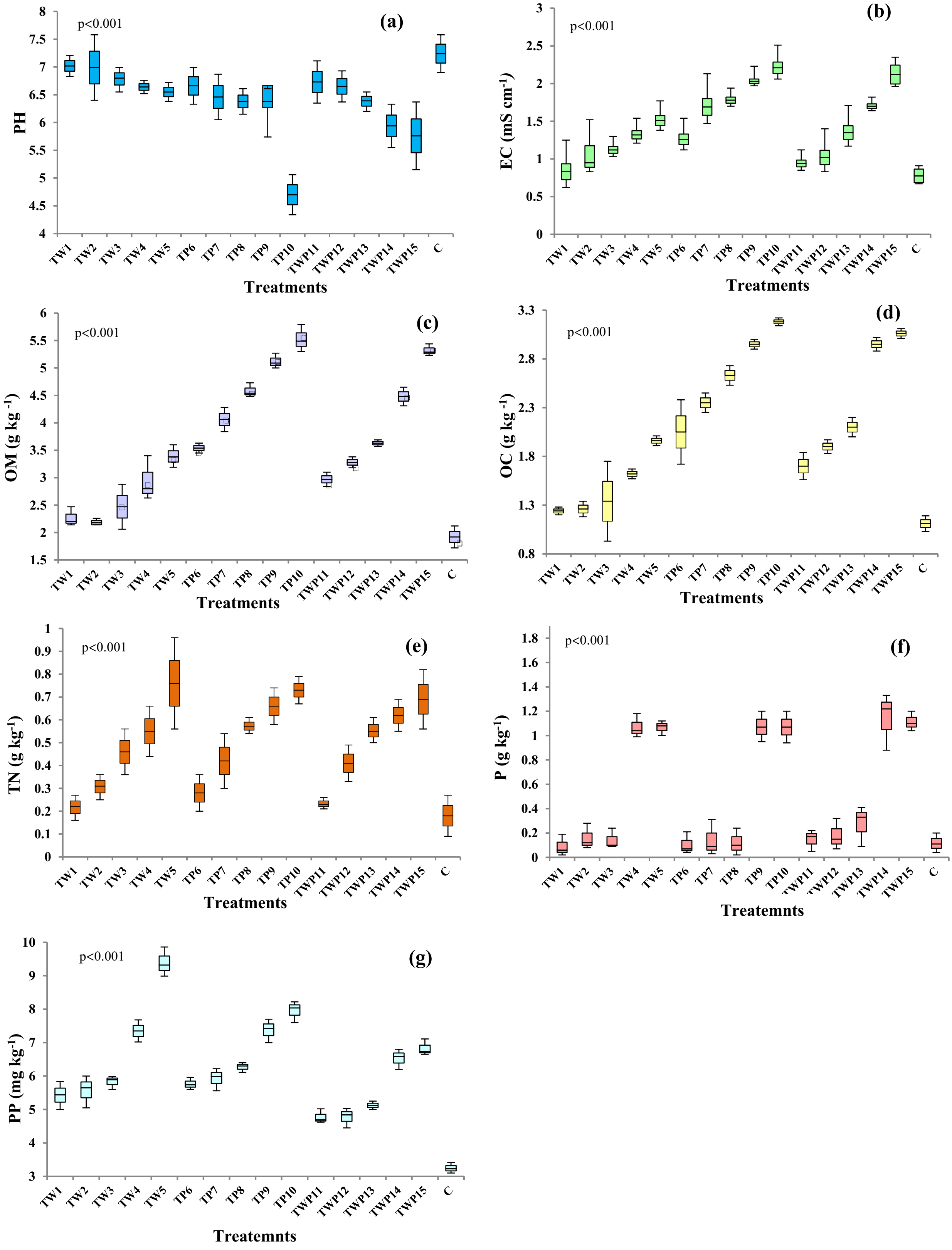

Fig. 1 Boxplots of the effects of OMWW and OMP and the Combination on the soil properties with *p* values showing the significant effects revealed by ANOVA. **a** pH, **b** *EC* electrical conductivity mS cm^{-1}; **c** *OM* organic matter g kg^{-1} DM, **d** *OC* organic carbon g kg^{-1} DM; e TN total nitrogen g kg^{-1} DM; **f** *P* assimilable phosphorus g kg^{-1} DM; **g** *PP* phenolic compounds mg kg^{-1} DM, TW1, TW2, TW3, TW4, TW5, TP6, TP7, TP8, TP9, TP10, TWP11, TWP12, TWP13, TWP14, TWP15: treatments, *C* control

noted that the soil EC changed as a function of the amount of OMP applied. The combination treatment showed a similar pattern of increase in soil EC as OMP.

Effect of OMWW, OMP, and the combination on soil OM

A comparison of the average OM content indicated the presence of 5 groups. Determination of soil organic matter showed that the control group initially had low organic matter content (Table 4). The increase in soil organic matter was very remarkable in the treatment with OMP and in the combination treatment (Fig. 1c). The OMWW treatment also modified the content of the soil OM. The recommended dose of OMWW resulted in an increase in soil OM, but this increase was not significant compared to the high doses of OMWW treatments. Furthermore, the increase in soil organic matter is proportional to the dose applied. The main reason for this increase is the high quantity of organic matter present in OMWW and OMP. The high amount of organic matter in the soil increases water retention capacity and improves soil stability by forming colloidal complexes with clay that can retain water [39]. Our results confirm previous reports [3, 11, 17, 40] in which OMWW had a positive effect on soil organic matter over both a short and long period of time. Furthermore, our results are in agreement with those of Ameziane et al. [22] and Aranda et al. [41], who found that amendment of soil with OMP increases soil organic matter and thus reduces soil susceptibility to erosion by increasing water retention capacity.

Effect of OMWW, OMP, and the combination on soil organic carbon (OC)

The contribution of olive mill waste treatment to soil organic matter increased the soil organic carbon content. The organic carbon content in different soil samples was correlated with the value of soil organic matter. In fact, the analysis of variance revealed a highly significant increase in soil organic carbon as a function of applied treatment compared to the control (Table 4 Fig. 1d). The positive effect of OM wastes on soil fertilization is in agreement with all previous studies on the effect of OMWW on soil organic matter [3, 17, 20, 42, 43]. In addition, Ameziane et al. [22] have demonstrated that amendment of soil with OMP significantly increased soil organic carbon content in a dose–response manner. Moreover, García-Ruiz et al. [44] and Aranda et al. [41] found that long-term application of OMP as a co-compost increased soil organic carbon content.

Effect of OMWW, OMP, and the combination on soil total nitrogen (TN)

A comparison of the average TN indicated the presence of 2 groups. The application of OMWW, OMP and the combination caused a slight modification in total nitrogen in soil (Fig. 1e). Analysis of variance showed a significant effect of the treatments applied on total nitrogen in the soil. In fact, the control soil was initially very poor on total nitrogen. The application of OMWW and OMP to the soil resulted in a slight increase in total nitrogen in the soil (Table 4). Furthermore, the combination also showed a slight increase compared to the control (Table 4). This is probably due to the dynamics of the element. All previous research found an increase in total nitrogen in the soil under treatment with OMWW and OMP (citation). This increase enhances the plant production as nitrogen is the pivot element for fertilization. However, a few studies reported a decrease or no significant changes in total nitrogen in soil during the short period of exposure to olive mill waste [17, 45, 46].

Effect of OMWW, OMP, and the combination on soil assimilable phosphorus (P)

A comparison of the average P values showed the presence of 2 groups. As for the phosphorus content of the soil, no significant effect was found at low doses, including the recommended doses for OMWW and OMP (Table 4) (Fig. 1f). These values classify the soil as very poor in phosphorus. After a three-year treatment with raw OMW, Chartzoulakis et al. [24] found no improvement in soil phosphorus content. Magdich et al. [38] found no significant effect on soil phosphorus under OMWW application, even at the three levels of OMWW (50, 100 and 200 m^3 per $year^{-1}$). This is most probably due to the immobilization of the element caused by the soil humic characteristic. Dakhli et al. [47] also found that OMWW had no effect on assimilable phosphorus in soil. Regarding the effect of OMP, Ameziane et al. [22] found that its amendment did not significantly increase the phosphorus content in the soil, mainly due to the low phosphorus content in OMP. In contrast, soils treated with the highest doses (75% and 100%) of OMWW and OMP, as well as the combination, showed a significant increase in soil assimilable phosphorus. Our results are in agreement with those of Kavvadias et al. [48] who found an increase in available phosphorus in the soil after treatment with raw OMWW. Furthermore, Di Bene et al. [15] reported an increase in total phosphorus and available phosphorus in soil after 5 days of application of 80 m^3 ha^{-1} OMWW. A recent study by Zema et al. [17] indicated that OMWW increases soil phosphorus content, thus improving soil fertility, and that the use of OMW reduces the need for chemical fertilizers, with obvious economic and environmental benefits.

Effect of OMWW, OMP, and the combination on soil phenolic compounds (PP)

Phenolic compounds are among the principal factors responsible for the toxicity of OMWW and OMP due to their phytotoxic and antibacterial properties as well as their toxic effects on soil invertebrates [49]. A comparison of the average PP indicated the presence of 6 groups. Analysis of variance showed that the OMWW and OMP treatments and the combination highly significantly increased the content of phenolic compounds in the soil compared to the control. This increase is proportional to the dose of olive mill waste added to the soil. The results showed an increase in phenolic compounds in the soil under the treatment with OMWW, OMP and the combination. The legally recommended doses increased soil polyphenols compared to the untreated soil (Table 4 Fig. 1g). These findings are in accordance with previous studies demonstrating that the application of OMWW significantly increases phenolic compounds in soil [11, 17, 20, 48, 50]. Phenolic compounds, together with other organic compounds, play an important role in the formation of soils. Due to the phytotoxicity of polyphenol metabolites, their presence in high quantities could have an impact on soil functioning [11].

FTIR analysis of OMWW and OMP

FTIR analysis is considered one of the most effective qualitative analyses for monitoring the functional groups of organic wastes, such as OMWW and OMP. Through the molecular vibrations of the chemical or biological compounds present in the sample, FTIR analysis provides a unique signature of the chemical or biochemical substance present (torsions of the chemical bonds, bending and stretching) [51, 52]. Figure 2 shows the FTIR spectra of the OMWW and OMP samples. The common absorption of OWWW and OMP is between 3700 and 3000 cm^{-1}, which represents the O–H stretching vibration of alcohols, phenolic groups, carboxylic groups and the amide N–H functions hydrogen vibration. Several previous studies have found intense broadband absorption between 3000 and 3800 cm^{-1} in OMWW and OMP, corresponding to phenolic compounds [52–56]. Long-chain aliphatic methylene molecules containing CH, CH_2, and CH_3 groups are found in the two peaks at 2900 and 2800 cm^{-1} representing the long chain lipids [55–58]. The next peaks at the absorption bands of 1750–1700 cm^{-1}, 1650 cm^{-1}, 1450 cm^{-1} could be due to the stretching vibration C = O of the ketones, aldehydes, and carboxylic acid groups and esters functional groups. This feature shows the acidic nature of the OMWs based on previous studies where the same bands were found [52, 56, 59, 60]. The band at 1650 cm^{-1} represents the aromatic compounds due to the C = C stretching of the aromatic ring of quinone [52, 58, 61]. Moreover, the band at 1400 cm^{-1} is mainly due to the O–H stretching vibration of the phenolic compounds in OMWs and aromatic ethers [52, 60, 61]. Furthermore, the absorbances at 1100 cm^{-1} represent a C–O stretching corresponding to the structure of polysaccharide cellulose, confirming the presence of glucidic compounds.

A comparative analysis by FTIR of the effects of OMWW and OMP and the combination on the soil

The average FTIR spectra (Fig. 3) for the soil treated with OMWW, OMP, and the combination and for the different doses were analyzed. The spectral waves were mostly similar in shape for the OMP treatment and the combination, while the OMWW treatment also showed almost similar peaks between the different doses used. Numerous peaks can be differentiated corresponding to the functional groups of the various components with the vibration mode. The bands at about 3700–3620 cm^{-1} were present in almost all treatments, even in the controls, except TP10 and TWP15 treatments, which correspond to the O–H stretching of clay minerals

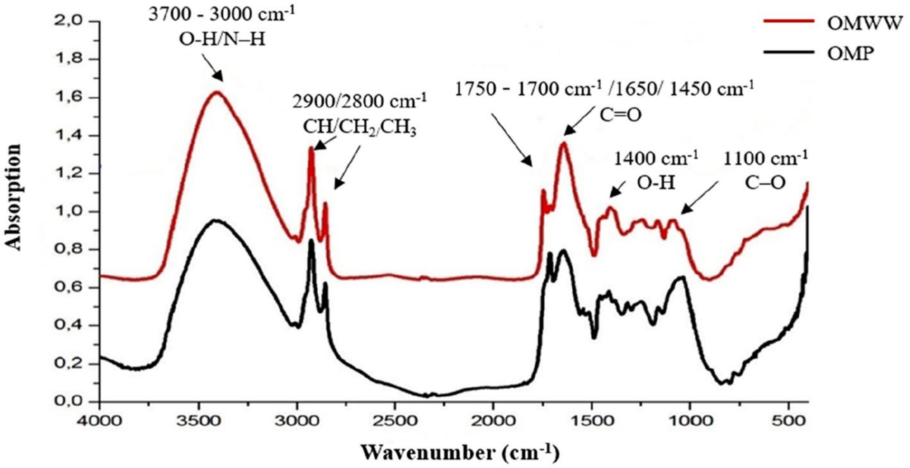

Fig. 2 FTIR spectra peaks of olive mill wastewater and olive mill pomace from 4000 to 500 (cm^{-1})

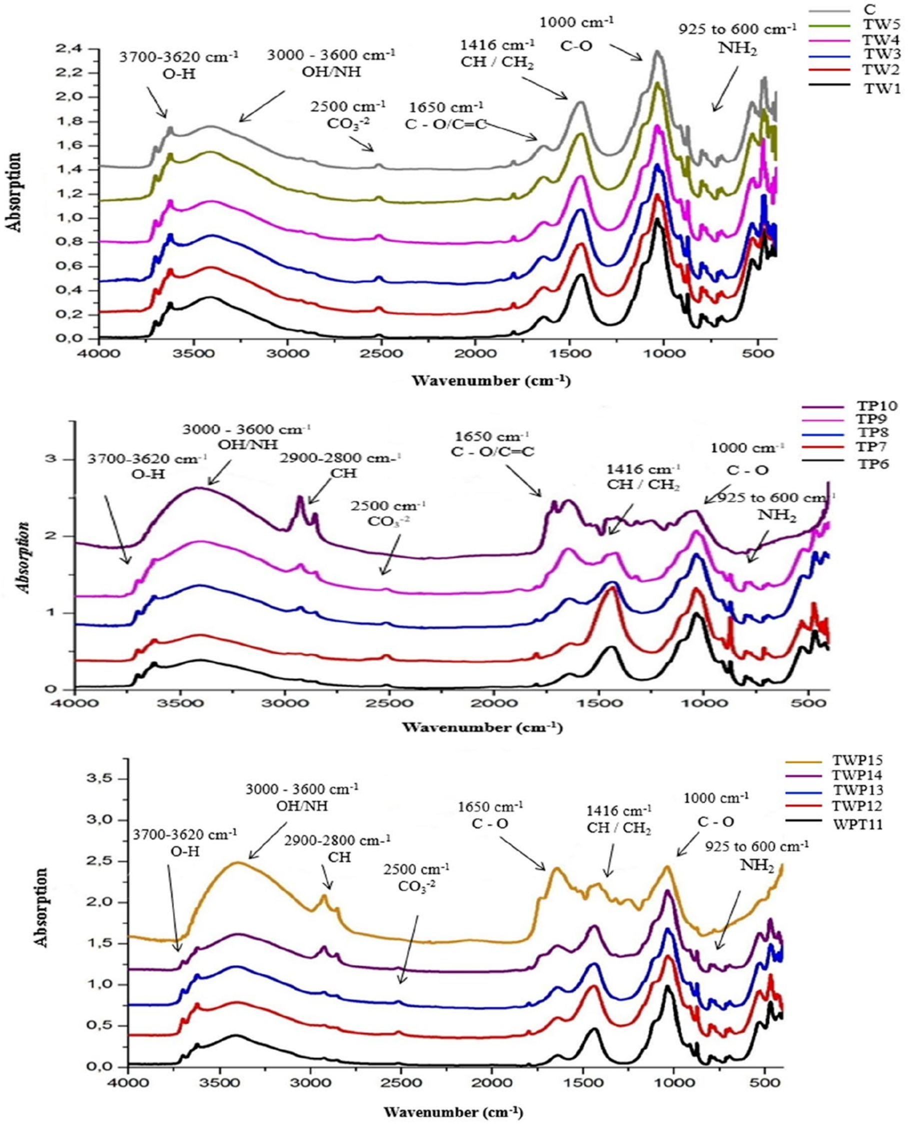

Fig. 3 FTIR spectra peaks of the soil treated with OMWW and OMP and the combination from 4000 to 500 (cm^{-1}). TW1: 12.5% OMWW, TW2: 25% OMWW, TW3: 50% OMWW, TW4: 75% OMWW, TW5: 100% OMWW, TP6: 12.5% OMP, TP7: 25% OMP, TP8: 50% OMP, TP9: 75% OMP, TP10: 100% OMP, TWP11: 12.5% Combination, TWP12: 25% Combination, TWP13: 50% Combination, TWP14: 75% Combination, TWP15: 100% Combination, *C* Control

Table 5 Correlation matrix (Pearson rank correlation) among the physicochemical parameters of the treated soil with OMWW and OMP and the combination with significance probability

	pH	EC	OM	OC	TN	P	PP
pH	1.000	< 0.0001	< 0.0001	< 0.0001	0.0013	0.0095	0.0254
EC	– 0.845	1.000	< 0.0001	< 0.0001	< 0.0001	0.0023	0.0060
OM	– 0.855	0.957	1.000	< 0.0001	0.0007	0.0136	0.0383
OC	– 0.851	0.943	0.992	1.000	0.0008	0.0099	0.0419
TN	– 0.730	0.847	0.754	0.749	1.000	0.0001	0.0004
P	– 0.626	0.704	0.602	0.623	0.813	1.000	0.0011
PP	– 0.556	0.654	0.522	0.513	0.778	0.739	1.000

EC electrical conductivity, *OM* organic matter, *OC* organic carbon, *TN* total nitrogen, *P* assimilable phosphorus, *PP* phenolic compounds

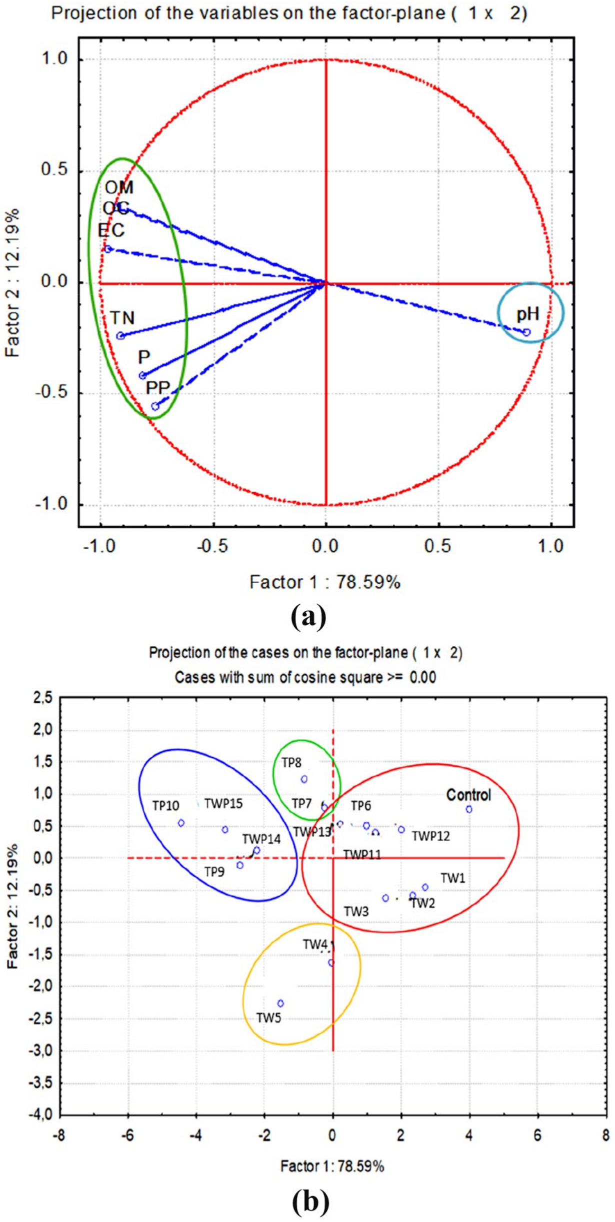

Fig. 4 Principal component analysis applied to soil properties treated with OMWW and OMP and the combination. a Correlation circles of the physico-chemical parameters of the soil pH, *EC* (electrical conductivity), *OM* (organic matter), *OC* (organic carbon), *TN* (total nitrogen), P (assimilable phosphorus), PP (phenolic compounds). b Projection of experimental points according to the treatment applied TW1: 12.5% OMWW, TW2: 25% OMWW, TW3: 50% OMWW, TW4: 75% OMWW, TW5: 100% OMWW, TP6: 12.5% OMP, TP7: 25% OMP, TP8: 50% OMP, TP9: 75% OMP, TP10: 100% OMP, TWP11: 12.5% Combination, TWP12: 25% Combination, TWP13: 50% Combination, TWP14: 75% Combination, TWP15: 100% Combination, Control untreated soil

[62]. Additionally, their absence in the 100% OMP treatment and the 100% of combination are mainly due to the high content of OMW in these treatments and the low content of the soil. The large absorption peak extending from around 3600 to 3000 cm^{-1} in all treatments, especially at high doses except for the untreated soil, is related to the OH stretching and/or NH stretching of the carboxyl, alcohol, amine and amide, hydroxyl groups, and phenolic compounds [58, 62, 63]. The presence of the peak at 3700 to 300 cm^{-1} previously found in the spectra of OMWW and OMP confirms that olive mill waste has increased content of phenolic compounds in the soil. These results are in agreement with those of Monetta et al. [64], who confirmed that soil amendment with OMW causes a significant increase in phenolic compounds in soil. Moreover, Dakhli et al. [47] confirmed that application of OMWs to soil causes a significant increase in phenolic compounds in soil. The two peaks at 2900–2800 cm^{-1} in some treatments (TP8, TP9, TP10, TWP14 and TWP15) correspond to the CH stretching of aliphatic methyl and methylene groups [58], which is probably due to the high content of soil organic matter in the OMW treatment with OMP and the combination, while the absence of aliphatic bands in the other treatments is probably due to the degradation of organic matter. The peak at 2500 cm^{-1} is attributed to the CO_3-$_2$ vibration of carboxylic acids and carbonates, although they have low absorbance [65]. We found this peak only in the control, the treatment with OMWW and the low doses of OMP and the combination. On the other hand, the application of OMP and the combination with the high doses (TP10 and TWP15) renders the soil not calcareous. Moreover, the band at 1650 cm^{-1}, which appears in almost all treatments, is associated with the C–O stretching and the aromatic C=C groups of the amides [66]. Furthermore, the shoulder band at 1416 cm^{-1} probably reflects the CH or CH_2 bending vibrations of the methyl groups [62]. Moreover, the peaks at 1000 cm^{-1} present in all treatments reflect the Si–O stretching and C–O bending of silicates and the polysaccharides of soil [67]. The successive bands from 925 to 600 cm^{-1} are attributed to Al–OH stretching and NH_2 vibration of kaolinite, primary amines and iron oxides, respectively [62, 67].

Correlation analysis

Correlations between the different pairs of traits are conducted to identify those traits that evolve in the same direction and those that develop in the opposite direction. Soil pH shows significant negative correlation with EC, OM, OC, TN, P, and PP. While the correlations between EC and OM, OC, TN, P, and PP are significantly positive (Table 5).

Multivariate analysis (PCA)

Principal component analysis was carried out to assess the trend of the various parameters studied (pH, EC, OM, OC, TN, P, and PP). The PCA (Fig. 4a) indicated the presence of two groups. The first group consists of pH, which showed positive correlations with axis 1 representing 78.59% of the information, while axis 2 explained 12.19% of the information. The second group comprises OM, OC, EC, TN, P, and PP, which showed negative correlations with axis 1. Interesting conclusions can be obtained from the multivariate statistical PCA analysis. First, OM, OC, EC, and TN, P, PP varied in the same direction due to the significant effect of the applied treatment. pH shows in the opposite direction in a single group, which is mainly due to the high content of EC, OM, OC, and PP in the soil caused by the applied treatments. In other words, the high content of all these parameters in the soil increases the acidity of the soil. Some of these associations are in agreement with those obtained by Chaari et al. [14] and Zema et al. [17] in their study. Specifically, those related to OM, TN, P, and PP. The PCA projection for the experimental points (Fig. 4b) showed that treatments TP8, TP9, TP10, TWP14, and TWP15 were negatively correlated with axis 1. These treatments include the high doses of OMP and the combination and recommended dose of OMP, which have a highly significant effect on soil parameters. The treatments TW1, TW2, TW3, TW4, TP6, TP7, TWP11, TWP12, and TWP13 and the control treatment indicated a positive association with both axes (1 and 2) except TW1, TW2, TW3, and TW4 including the allowed dose of OMWW which were negatively associated with axis 2. These treatments showed an effect on soil properties although this effect was not highly negative. Moreover, TW5, which corresponded to the 100% OMWW treatment, was negatively associated with axes 1 and 2, in the opposite direction as TP10 and TWP15, although all these treatments corresponded to 100%, mainly because TW5 did not show a highly negative effect on soil properties as did TP10 and TWP15.

Conclusion

To demonstrate safety and make the application more efficient, the current research focused on determining the potential effects of different concentrations of OMWW and OMP, including recommended doses, and the effects of the combination on key soil properties. From the results of this study, we can conclude that:

- Treatment of soil with various concentrations of OMWW, OMP, and the combination had a significant effect on soil physicochemical properties. A strong increase in the concentrations of several mineral elements was observed, as well as a significant increase of organic matter in the soil, especially at high doses.
- Comparing the effects of olive mill waste, the results showed similar effects of treatment with OMWW and OMP and the combination on soil properties in most cases. A dose–response effect was found for most of the parameters studied. The effects on soil fertility were stronger after treatment with OMP and the combination than for treatment with OMWW.
- It is worth highlighting the benefits of using FTIR to characterize the changes in soil properties and phenolic compounds in the soil after treatment with olive mill waste, especially for the phenolic compounds in the soil. This qualitative analysis can be done quickly and relatively inexpensively.
- This study recommends a significant dilution of olive mill waste as a possible solution before its disposal, in addition to avoid risks to the soil and to facilitate the safe and environmentally friendly use of these by-products. In addition, this study recommends the application of these by-products to the soil at a dosage of 12.5% to 25% (w/w) for OMWW and OMP, as well as the combination treatment based on the results obtained in this study.

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Data availability All data generated or analysed during this study are included in this published article.

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