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# Long-term changes in soil properties according to different strategies of ferti-irrigation with olive oil mill wastewater in olive groves of a Mediterranean region

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## Abstract

The effects of application of olive mill wastewaters (OMW) has been mainly studied in the short-term, while the literature about its impacts after many years has been much lower. This study has monitored some soil physico-chemical parameters after 20 years from OMW application. Two OMW application strategies were tested: intermittent irrigation (i.e., every two years) and continuous irrigation (i.e., each year), both at a dose of 50 $m^3$/ha per year; a non-irrigated soil was assumed as control, since never treated with OMW. Comparisons between long-term and short-term changes (from a previous investigation) were also carried out for key soil properties.

In comparison to the control sites, all physico-chemical properties of soils treated with OMW significantly changed, regardless of the irrigation strategy. Noticeable increases were measured for soil salinity (up to +70%) and content in polyphenols (+120%), which suggests paying attention to avoid degradation in soil quality. These effects were lower in the case of intermittent irrigation. The study also evidenced that some short-term undesired effects of OMW application decreased several after irrigation (e.g., increase in soil pH and salinity) down to tolerable values. Therefore, it can be concluded that the annual or inter-annual applications of OMW make the soil fertility stable or even increase it in the short term, but intermittent irrigation is advisable to avoid undesired impacts for crops and ecosystem.

**Key words:** OMW application; soil salinisation; ; soil organic matter; soil acidification; polyphenols.

## 1 Introduction

Oil extraction from olives produces solid ("husk" or "pomace") and liquid (olive oil mill wastewater, hereafter "OMW") residues. OMW consists of oil emulsion, process water, and olive pulp fragments (Yamani et al., 2020), and its physico-chemical characteristics depend on the oil extraction techniques and physiological parameters of olives (Barbera et al., 2014; Mekersi et al., 2021). The main physico-chemical characteristics of OMW are a very high acidity and noticeable concentrations of various organic (e.g., organic acids, pectins and polyphenols) and inorganic compounds or elements (e.g., sulphates, chlorides, phosphoric salts, potassium and sodium) (Jamrah et al., 2023). Due to these intrinsic characteristics, OMW has a high pollution potential for soil and water bodies due to the uncontrolled disposal in the environment (Foti et al., 2021; Regni et al., 2021). These pollution hazards require proper techniques for OMW management, such as the treatment in municipal plants or other physico-chemical or biological treatments (Dourou et al., 2016). However, these techniques are complex and expensive, and their depuration efficiency is not always high (Calabrò et al., 2018). A viable alternative to manage OMW with cheapness and environmental safety is the use of OMW for ferti-irrigation of olive groves, which has been proposed for a long time to increase soil fertility and crop productivity. At the same time, this practice, improves the rational use of natural resources and promotes the principles of the circular economy (Gargouri et al., 2022), especially in semi-arid croplands.

The beneficial effects of OMW land spreading on agricultural soils are well known for decades. OMW application increases the contents of organic matter and nutrients (nitrogen, phosphorus, and especially potassium) in the treated soils (Chaâri et al., 2022; Mahmoud et al., 2022). Moreover, water infiltration and stability of aggregates of soils improve after irrigation with OMW (Albalasmeh et al., 2019; Bombino et al., 2021), and these effects result in increased water content and oxygen storage. However, despite these positive effects of OMW application on agricultural soils, several negative impacts are possible. For instance, the very low pH of OMW may increase the soil acidity (Di Serio et al., 2008), and the high content of organic matter and relatively low concentration of nitrogen may unbalance the C/N ratio of soil (Barbera et al., 2014; Piotrowska et al., 2011). Moreover, the presence of inhibiting compounds that are toxic for the microbiota of soil and crops, such as the polyphenols, may result in irreversible effects on soil health and crop growth (Mekki et al., 2007). These undesired effects suggest carefully monitoring the most important physico-chemical parameters of soil, such as the electrical conductivity, pH, organic matter, nutrients, C/N ratio, ions and polyphenols, directly impacted by the specific characteristics of OMW.

The changes in soil properties after OMW application may be transient (i.e., they disappear some years after irrigation stops) or even permanent. The short-term investigations highlight the immediate response of soil to this practice, but do not allow disentangling the permanent or long-lasting impacts. Therefore, there is a need to check how long soil health and productivity are affected by this practice many years after wastewater application.

The scientific literature has widely explored the impacts of OMW application that strictly depend on the specific characteristics of treated soils and cultivated crops. For instance, a few years after OMW application on soil, Yamani et al. (2020) and Mekersi et al. (2022) found increases in organic load, polyphenols and salt concentration, but no significant effects on phosphorus or total nitrogen, while, more recently, Khalil et al. (2024) detected improvements in contents of macro- and micronutrients that were proportional to the amount of OMW applied but resulted in imbalance in soil nutrients. However, compared to numerous short-term investigations, few studies have analysed the long-term effects of OMW land spreading on olive groves, which may reveal whether the impacts of this practice are durable and affected by the superimposition effect due to the repeated applications – that is, year by year - of OMW on soil.

Therefore, there is an evident need for further research in the long term, especially in croplands where, due to the specific agro-climatic conditions, soil health and crop productivity can be irreversibly affected by the detrimental impacts of this practice. Since these effects are strictly associated with the specific characteristics of the treated soils and the growing crops, their response to OMW application may be contrasting and quite controversial, and their verification is essential to propose a safe and profitable practice for wastewater disposal and soil fertilisation to farmers and agronomists.

To fill these gaps, this study has evaluated the main physico-chemical properties of soil irrigated with OMW throughout 20 years in an olive grove in Tunisia. Two irrigation strategies have been investigated, that is an intermittent (each two years) and an annual irrigation using OMW. The research questions that have inspired this research are: (i) Do the studied physico-chemical properties of the treated soils significantly change between these irrigation strategies and untreated soil? and (ii) What are the changes in the main physico-chemical properties in the long term compared to the short term, the latter measured in a parent study carried out in the same site about 20 years ago? This study aims to provide valuable information on the response of the rhizosphere after long-term ferti-irrigation with OMW using a dataset of meaningful indicators of soil quality.

## 2 Materials and Methods

### 2.1 Study area

The investigation at the Taous experimental station of the Olive Tree Institute ("Taous") in the region of Sfax, Tunisia, North Africa (34°43'N, 10°41'E) (Figure 1). This region is characterized by a typical Mediterranean climate, Csa type according to the Koppen classification (Kottek et al., 2006). The mean rainfall is 200 mm per year, and the annual average temperature is 23 °C. The soil has a sandy-silty texture (Table 1).

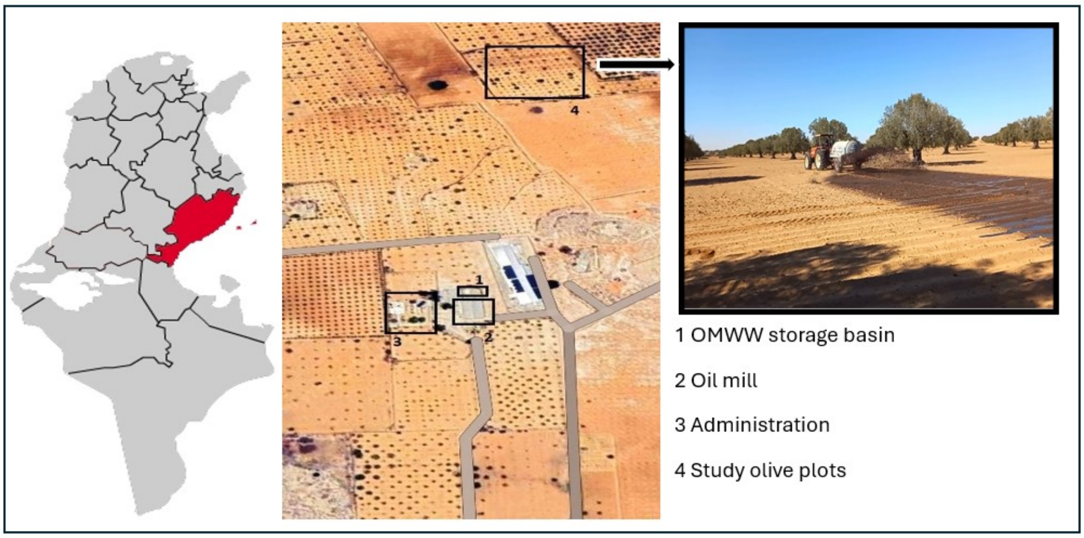


Figure 1 - Geographical location of and aerial map of the study area (Taous, Sfax region, Tunisia).

Table 1 – Analysis of soil texture (mean ± standard deviation, n = 3 plots) of the experimental olive grove (Sfax, Tunisia).

| **Soil content (%)** | **Irrigation with OMW** | | | **Irrigation with clear water (control)** | | |
|---|---|---|---|---|---|---|
| | *Soil depth (cm)* | | | | | |
| | 0–30 | 30-60 | 60-90 | 0-30 | 30-60 | 60-90 |
| Sand | 83.1 ± 2.62 | 80.2 ± 1.1 | 79.16 ± 1.8 | 81.1 ± 0.50 | 79.1 ± 1.8 | 76.9 ± 1.6 |
| Clay | 0.17 ± 0.05 | 0.18 ± 0.02 | 0.13 ± 0.03 | 0.20 ± 0.01 | 0.13 ± 0.03 | 0.15 ± 0.04 |
| Silt | 16.7 ± 2.64 | 19.6 ± 1.11 | 20.7 ± 1.8 | 18.7 ± 0.49 | 20.8 ± 1.83 | 23 ± 1.12 |

Sfax has a semi-arid to arid climate, receiving on average 200 mm of rain per year and showing a mean temperature of 19.6 °C (last 60 years of historical data). Both weather variables show a very low variability over time, and this is mainly in the five years of the monitoring (2005-2007 and 2022-2023). In these years, the variability of weather was even lower than in the entire period (mean temperature in the range of 20 to 21.3 °C and annual precipitation in the range of 106 to 191 mm, the latter being much lower than the saturation content of the soil), which ensures that the meteorological effects throughout the observation periods were negligible.

## 2.2 Experimental design

In this area, an experimental grove with 80-year-old olive trees (*Olea europea* L. var. *Chemlali*) was selected in 2004. In this site, three groups of plots were delimited (1-hectare area for each one), of which the first group of three plots were treated with OMW from 2005 to 2023 every two years (not irrigated in 2022, hereafter indicated "No-irr"), a second group was irrigated with OMW until 2023 (dose of 50 $m^3$/ha, "Irr"), and a third group of three plots (assumed as control, "C") was never treated with OMW. Therefore, two irrigation strategies were investigated, of which the first strategy is intermittent irrigation (every two years), and the second strategy is annual irrigation, both using OMW. The effects of these strategies on soil properties were compared to those measured in untreated soils, that is in olive groves never treated with OMW.

Finally, the effects of these strategies (long-term irrigation with OMW) on soil were compared to the short-term effects, using the data collected between 2005 and 2007 and previously published by Magdich et al. (2013), working in the same olive groves. About the semi-arid areas, it is common that short-term often refers to periods ranging from a few months to a very few years (no more than five), while long-term typically refers to periods extending beyond 10 years or much more (Sainju et al., 2022; Yang et al., 2022). Therefore, the investigations carried out in 2003-2005 (approx. 20 years from 2024) can be considered as "long-term" monitoring, while the observations made in 2021-2023 (1-2 years before 2024) are related to the "short-term" period. The weather conditions were comparable (mean precipitation of 152 and 134 mm/yr, and mean temperature of 20 and 21.4 °C) and the agro-pedological conditions were practically the same in the two periods.

Hereafter, the soil conditions to be compared will be referred to as follows: (i) short-term irrigation (ST/T, short-term treatment, LT/T/No-irr, long-term treatment with intermittent irrigation, LT/T/Irr with annual irrigation, always using OMW, and C, control, with data averaged among observation in 2005 to 2007, 2022 and 2023).

2.3 Olive mill wastewater characterization

The OMW used for irrigation was taken from a three-phase continuous extraction mill of Sfax. OMW samples were collected and stored in containers and carried to the laboratory to be directly analyzed: (i) pH and electrical conductivity (EC) were measured directly on the effluent using a Mettler Toledo MP 220 pH meter and a Mettler Toledo MC 226 EC meter, respectively according to a standard method (Sierra et al., 2007); (ii) dry matter (DM), by weighing the sample before and after drying overnight at 105 °C; (iii) Total Organic Carbon (TOC), estimated as the difference between dry matter and residue after calcinations at 550 °C for 4 hours; (iv) nitrogen (N), measured by the Kjeldahl method (Kandeler & Gerber, 1988); (iv) phosphorus (P), magnesium (Mg), potassium (K) and sodium (Na), determined by atomic absorption using Hitachi U-2000 spectrometer; (v) chloride, according to the Mohr method (Rodier et al., 2009); (vi) polyphenols (PP), using the Folin-Ciocalteu method (Singleton & Rossi, 1965); (vii) Chemical Oxygen Demand (COD), evaluated according to the standard method of Knechtel (1978); and (viii) Biochemical Oxygen Demand ($BOD_5$) over five days, determined by the manometric method with a respirometer (Aloui et al., 2007). All OWW characteristics are summarized in Table 2.

Table 2 – Main characteristics of OMW (mean ± standard deviation, n = 3 plots) used for irrigation of the experimental olive grove (Sfax, Tunisia).

| **Parameters** | **Irrigation season** | |
|---|---|---|
| | **2021** | **2023** |
| pH | 5.23 ± 0.16 | 5.10 ± 0.09 |
| EC (mS/cm) | 24.8 ± 0.31 | 25.3 ± 0.31 |
| DM (g/L) | 149 ± 0.64 | 150 ± 0.48 |

| | | |
|---|---|---|
| TOC (g/L) | 29.5 ± 0.13 | 26.4 ± 0.21 |
| COD (g/L) | 76 ± 0.23 | 95 ± 0.55 |
| $BOD_5$ (g/L) | 19 ± 0.07 | 39 ± 0.87 |
| N (g/L) | 1.70 ± 0.12 | 1.79 ± 0.07 |
| K (g/L) | 4.12 ± 0.31 | 5.89 ± 0.26 |
| Na (g/L) | 1.22 ± 0.16 | 1.4 ± 0.22 |
| P (g/L) | 0.50 ± 0.11 | 0.73 ± 0.08 |
| Cl (g/L) | 0.74 ± 0.05 | 0.69 ± 0.11 |
| Mg (g/L) | 0.49 ± 0.14 | 0.53 ± 0.05 |
| Ca (g/L) | 0.61 ± 0.06 | 0.58 ± 0.08 |
| PP (g/L) | 1.76 ± 0.07 | 1.81 ± 0.16 |

Notes: EC = electrical conductivity; DM = dry matter; TOC = Total Organic Carbon; TKN = Total Kjeldahl Nitrogen PP = polyphenols.

## 2.4 Soil sampling and analysis

Twelve samples of soil were taken in points randomly chosen in treated and control plots at two depths (0-30 and 60-90 cm) in 2022 in the “T/No-irr” plots, in 2023 in the “T/Irr” plots, and in 2022 and 2023 in the “C” plots. Sampling operations were manually carried out using a steel ring (0.1 m in diameter) to gently extract the core from the soil (after excavation for the deeper sampling depths). These samples were kept in plastic bags and brought back to the laboratory for subsequent analyses.

The following physico-chemical properties of soil were measured: (i) pH and EC, with pH-meter and conductivimeter in soil-water extract (1/2.5: w/v) (Paredes et al., 1987); (ii) Soil Water Capacity (SWC), using the traditional funnel method; (iii) Cation Exchange Capacity (CEC), by extraction with sodium acetate (Klute, 1986); (iv) OM, calculated from Total Organic Carbon (TOC), the latter measured by oxidizing the samples with potassium dichromate titration of $FeSO_4$ (Walkley & Black, 1934); (v) total Kjeldahl nitrogen (TKN), by Kjeldahl method (Kandeler & Gerber, 1988); (vi) phosphorus (P), by using the method of Olsen (1982); (vii) potassium (K) and sodium (Na), using a flame photometer (Jenway, PEP- 7); (viii) chloride (Cl), following the

Mohr method (Rodier, 2009); and (ix) polyphenols (PP), determined by using the Folin-Ciocalteu method (Waterman & Mole, 1994).

### 2.5 Statistical analysis

A one-way ANOVA followed by Tukey's pairwise comparisons (at $p < 0.05$) was used to identify statistical differences in the soil properties between three soil conditions (control, treated and non-irrigated, and treated and irrigated with OMW) at each soil depth (0-30, topsoil, and 60-90 cm, deeper soil), considered as an independent factor. +. The need to separately explore these differences at the two soil depths was justified by the significant differences in all soil properties between the two layers, revealed by a two-way ANOVA with soil condition and depth as factors.

Then, an agglomerative hierarchical cluster analysis (AHCA), a distribution-free ordination technique to group sites with similar characteristics by considering an original group of variables, to group soil samples with similar characteristics. As similarity-dissimilarity measure, the Euclidean distance has been used. WUA grouping through CA has been reported in a dendrogram.

Finally, another one-way ANOVA followed by Tukey's pairwise comparisons (at $p < 0.05$) was used to identify statistical differences in the soil properties between the short (data of 2022-2023) and long-term (2005-2007) irrigation under four soil conditions (control, treated in the long-term, treated and non-irrigated in the short term, and treated and irrigated with OMW in the short term) considered as an independent factor. In this case, the observations made at different soil depths were combined. All statistical analysis was conducted using the XLSTAT software (release 2019, Addinsoft, Paris, France).

## 3 Results

### 3.1 Effects of long-term irrigation with OMW on soil properties

ANOVA showed that the soil condition significantly changed all soil properties at both depths except C/N in the topsoil (Table 3).

Table 3 - Results of one-way ANOVA applied to the soil properties measured at two depths (0-30 and 60-90 cm) under three soil conditions (control, intermittent irrigation and permanent irrigation with OMW) in the experimental plots (Sfax, Tunisia).

| **Soil properties** | **Sum of squares** | **Mean squares** | **F** | **Pr > F** | **Sum of squares** | **Mean squares** | **F** | **Pr > F** |
|---|---|---|---|---|---|---|---|---|
| | *0 - 30 cm* | | | | *60 - 90 cm* | | | |
| pH | 0.019 | 0.010 | 6.198 | **0.020** | 0.01 | 0.01 | 10.13 | **0.00** |
| EC | 21900 | 10950 | 723.762 | **<0.0001** | 3563 | 1781 | 100.94 | **<0.0001** |
| OM | 25.895 | 12.948 | 299.508 | **<0.0001** | 11.34 | 5.67 | 969.85 | **<0.0001** |
| CEC | 1.147 | 0.573 | 415.637 | **<0.0001** | 0.73 | 0.37 | 129.83 | **<0.0001** |
| SWC | 68.500 | 34.250 | 21.759 | **0.000** | 129.00 | 64.50 | 29.03 | **0.00** |
| TN | 0.132 | 0.066 | 95.728 | **<0.0001** | 0.05 | 0.03 | 146.41 | **<0.0001** |
| C/N | 2.091 | 1.045 | 3.811 | 0.063 | 1.16 | 0.58 | 5.32 | **0.03** |
| P | 0.000 | 0.000 | 32.472 | **<0.0001** | 0.00 | 0.00 | 8.07 | **0.01** |
| K | 0.006 | 0.003 | 30.568 | **<0.0001** | 0.00 | 0.00 | 34.48 | **<0.0001** |
| Na | 0.002 | 0.001 | 8.250 | **0.009** | 0.00 | 0.00 | 14.25 | **0.00** |
| Cl | 0.006 | 0.003 | 18.389 | **0.001** | 0.00 | 0.00 | 30.68 | **<0.0001** |
| PP | 0.105 | 0.052 | 24.758 | **0.000** | 0.02 | 0.01 | 35.33 | **<0.0001** |

Notes: EC = electrical conductivity; SWC = Soil Water Capacity; CEC = Cation Exchange Capacity; OM = organic matter; TKN = Total Kjeldahl Nitrogen; PP = polyphenols; values in bold are significant at $p < 0.05$.

A detailed analysis of changes in the properties of the surface layer among the three soil conditions reveals that pH was significantly different between the C (8.81 ± 0.02), and treated plots (8.72 ± 0.03, T/No-irr, and 8.74 ± 0.08, T/Irr). The lowest (154 ± 2.86 μS/cm) and highest (256 ± 5.03 μS/cm) EC were measured in C and T/Irr plots, and both values were significantly different compared to T/No-irr soils (164 ± 4.73 μS/cm). CEC significantly increased after the treatments (3.81 ± 0.04 cmol/$kg_{DM}$, T/No-irr, and 3.74 ± 0.04 cmol/$kg_{DM}$, T/Irr) in comparison to C (3.16 ± 0.04 cmol/$kg_{DM}$). The SWC significantly increased only in the T/Irr plots (515 ± 1.53 g/$kg_{DM}$), while the differences between C (509 ± 0.98 g/$kg_{DM}$) and T/No-irr (510 ± 1.53 g/$kg_{DM}$) were not significant. A significant gradient C < T/No-irr < T/Irr in OM (8.34 ± 0.27 g/$kg_{DM}$, 9.36 ± 0.12 g/$kg_{DM}$ and 11.9 ± 0.04 g/$kg_{DM}$), in TN (0.45 ± 0.02 g/$kg_{DM}$, 0.55 ± 0.02 g/$kg_{DM}$ and 0.71 ± 0.05 g/$kg_{DM}$) and in P (0.023 ± 0.001 g/$kg_{DM}$, 0.028 ± 0.002 g/$kg_{DM}$ and 0.029 ± 0.001 g/$kg_{DM}$) was noticed. Also PP followed the same gradient, this parameter being 0.18 ± 0.06 g/$kg_{DM}$ in C, 0.31 ± 0.02 g/$kg_{DM}$ in T/No-irr, and 0.4 ± 0.01 g/$kg_{DM}$ in T/Irr. The C plots showed the lowest K (0.15 ± 0.01 g/$kg_{DM}$), and the T/Irr the highest (0.20 ± 0.01 g/$kg_{DM}$), both values being significantly different compared to the T/No-irr soils (0.18 ± 0.01 g/$kg_{DM}$). Na and Cl were the minimum in the C soils (0.10 ± 0.01 g/$kg_{DM}$ and 0.08 ± 0.01 g/$kg_{DM}$, respectively), and the maximum in T/Irr plots (0.13 ± 0.01 g/$kg_{DM}$ for both conditions). However, the value of Na in the T/No-irr soils (0.12 ± 0.02 g/$kg_{DM}$) was significantly higher compared to the control and similar as Na of T/Irr, while Cl of those soils (0.09 ± 0.02 g/$kg_{DM}$) was similar as the control, and significantly lower compared to T/Irr soils (Figure 2).

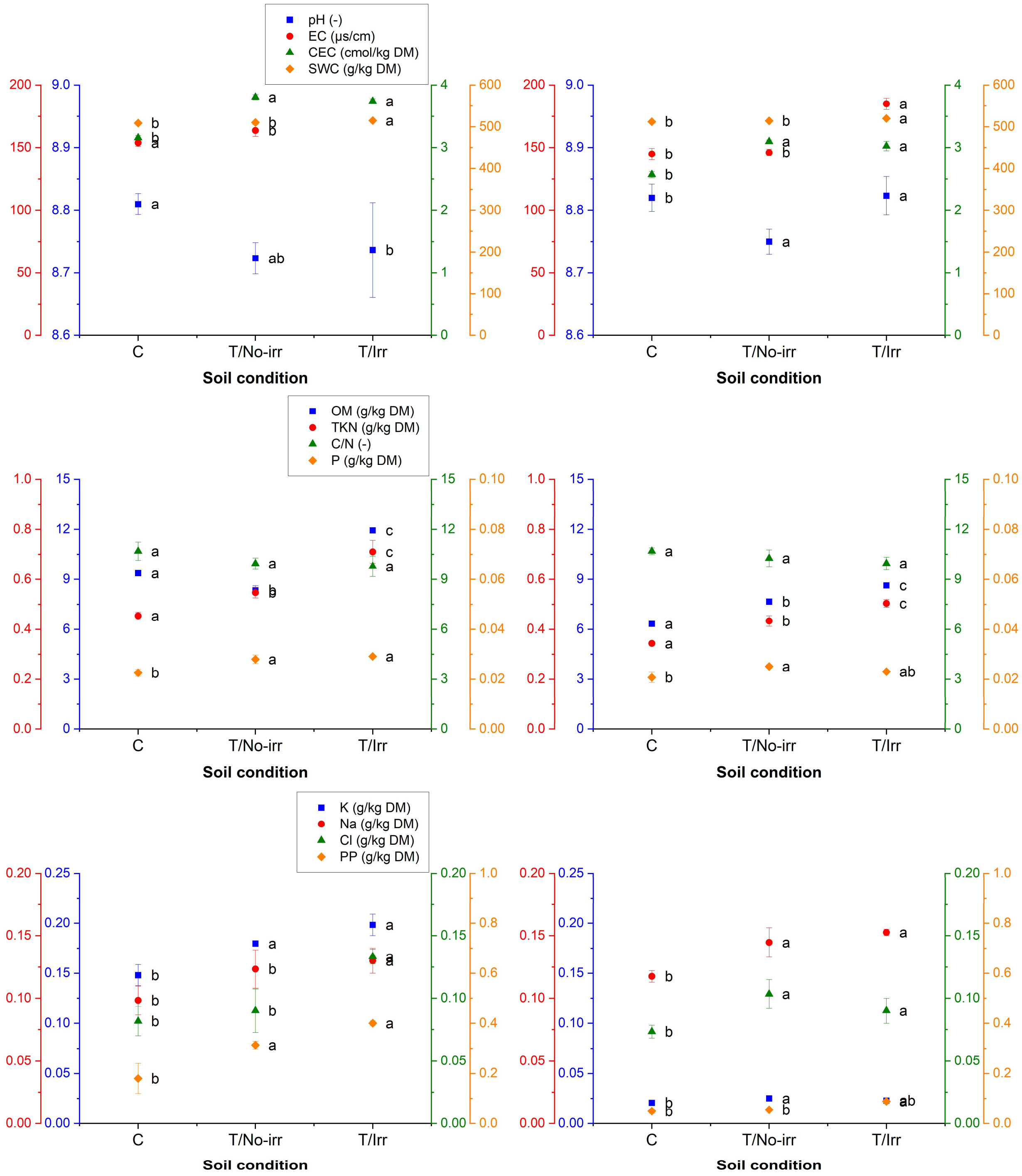


Figure 2 - Mean ± standard deviation (n = 9) of soil properties measured at two depths (0-30 cm, left panels, and 60-90 cm, right panels) under three soil conditions (control - C, long-term treated with OMW and non-irrigated in 2022 – T/No-irr, and long-term treated with OMW and irrigated in 2022 and 2023 - T/Irr) in the experimental plots (Sfax, Tunisia).

*Legend: Notes: EC = electrical conductivity; SWC = Soil Water Capacity; CEC = Cation Exchange Capacity; OM = organic matter; TKN = Total Kjeldahl Nitrogen; PP*

*= polyphenols. Different letters indicate significant differences after Tukey's test at p < 0.05.*

In the deeper layer, pH was similar in the C and T/Irr plots (8.82 ± 0.02 or ± 0.03), and these values were significantly higher compared to the T/No-irr soils (8.75 ± 0.02). EC, equal to 145 ± 4.54 µS/cm in C soils and 146 ± 2.65 µS/cm in T/No-irr plots), significantly increased in T/Irr plots (185 ± 4.58 µS/cm) and these soils also showed a significant increase in SWC (520 ± 1 g/kg$_{DM}$) compared to C (512 ± 1.79 g/kg$_{DM}$) and T/No-irr plots (514 ± 1 g/kg$_{DM}$), the latter values being statistically similar. The lowest CEC was measured in the C plots (2.57 ± 0.05 cmol/kg$_{DM}$), a value significantly lower compared to T/Irr (3.02 ± 0.08 cmol/kg$_{DM}$) soils, the T/No-irr sites showing intermediate CEC (3.1 ± 0.03 cmol/kg$_{DM}$).

OM, TN and P were significantly higher in the treated plots (7.66 ± 0.10 g/kg$_{DM}$, 0.43 ± 0.02 g/kg$_{DM}$, and 0.023 ± 0.001 g/kg$_{DM}$, T/No-irr, and 8.63 ± 0.07 g/kg$_{DM}$, 0.50 ± 0.02 g/kg$_{DM}$, and 0.025 ± 0.001 g/kg$_{DM}$, T/Irr) compared to the control (6.33 ± 0.07 g/kg$_{DM}$, 0.34 ± 0.01 g/kg$_{DM}$ 4, and 0.021 ± 0.002 g/kg$_{DM}$, respectively). The C/N ratio was significantly different only between C (10.7 ± 0.20) and T/Irr (9.95 ± 0.38) soils. The treated soils showed significant increases in K (0.14 ± 0.01 g/kg$_{DM}$, T/No-irr, and 0.15 ± 0.01 g/kg$_{DM}$, T/Irr) compared to C plots (0.12 ± 0.01 g/kg$_{DM}$), while Na and Cl were significantly different between the T/Irr plots (0.10 ± 0.01 g/kg$_{DM}$ and 0.06 ± 0.01 g/kg$_{DM}$) or T/irr (0.09 ± 0.01 g/kg$_{DM}$ for both ions) and the corresponding control (0.07 ± 0.01 g/kg$_{DM}$ and 0.05 ± 0.01 g/kg$_{DM}$, respectively). Finally, the long-term application of OMW resulted in an increase in PP both in the T/No-irr (0.18 ± 0.01 g/kg$_{DM}$) and T/Irr (0.16 ± 0.03 g/kg$_{DM}$) compared to the untreated plots (0.09 ± 0.01 g/kg$_{DM}$) (Figure 2).

The AHCA groups the measurements of soil properties made under the three soils conditions in third homogenous clusters (Figure 3). In more detail, a first cluster (C1) consists of observations at T/Irr soils (top layer), a second (C2) groups T/irr soils (deeper layer), while the third cluster is composed by C and No-irr/T soils (both layers).

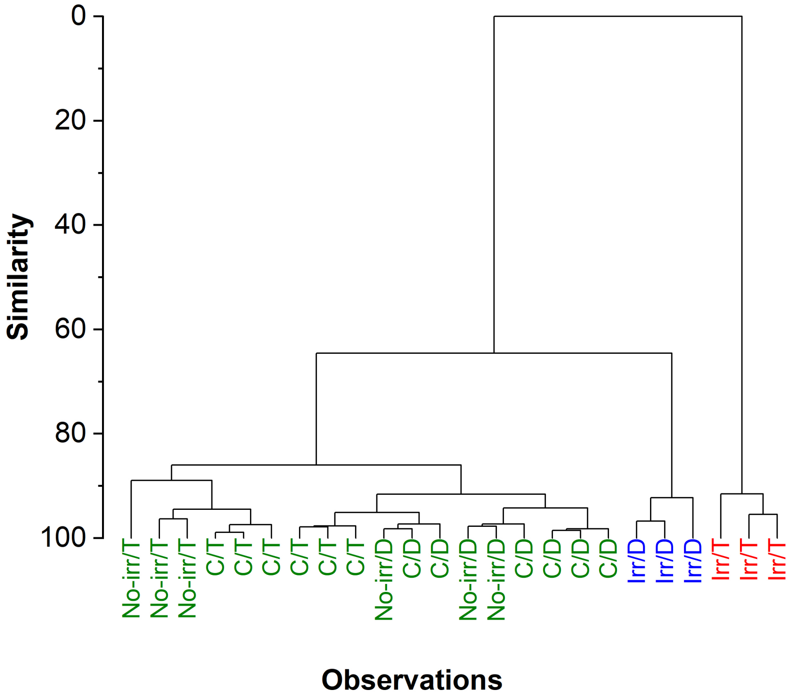


Figure 3 - Dendrogram provided by the Agglomerative Hierarchical Cluster Analysis of measured at two depths (0-30 and 60-90 cm) under three soil conditions (control - C, long-term treated with OMW and non-irrigated in 2022 - No-irr, and long-term treated with OMW and irrigated in 2022 and 2023 - Irr; D adter "/" stands for deeper layer, while T stands for topsoil layer) in the experimental plots (Sfax, Tunisia).

## 3.2 Effects of short-term and long-term irrigation with OMW on soil properties

According to ANOVA, all soil properties were significantly different between short-term and long-term irrigation (Table 4).

Table 4 - Results of one-way ANOVA applied to the soil properties measured under four soil conditions (control, treated in the short term, 2005-2007, long-term treated with OMW and non-irrigated in 2022, and long-term treated and irrigated with OMW in 2022 and 2023) in the experimental plots (Sfax, Tunisia).

| **Soil properties** | **Sum of squares** | **Mean squares** | **F** | **Pr > F** |
|---|---|---|---|---|
| pH | 0.093 | 0.031 | 31.708 | **<0.0001** |
| EC | 1307770 | 435923 | 19.144 | **<0.0001** |
| OM | 252.794 | 84.265 | 22.254 | **<0.0001** |
| TKN | 2.188 | 0.729 | 33.156 | **<0.0001** |
| P | 0.004 | 0.001 | 29.421 | **<0.0001** |
| K | 0.182 | 0.061 | 28.951 | **<0.0001** |
| Na | 0.093 | 0.031 | 31.708 | **<0.0001** |

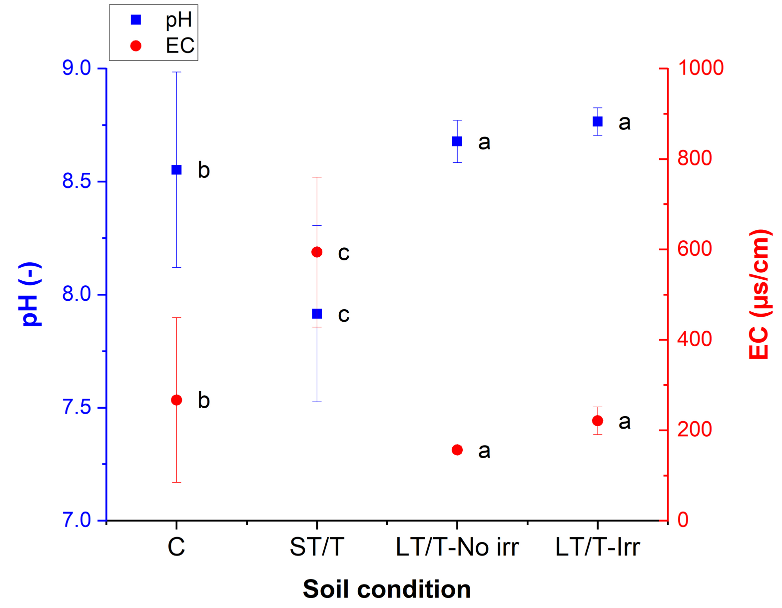
Note: EC = electrical conductivity; OM = organic matter; TKN = Total Kjeldahl Nitrogen; values in bold are significant at $p < 0.05$.

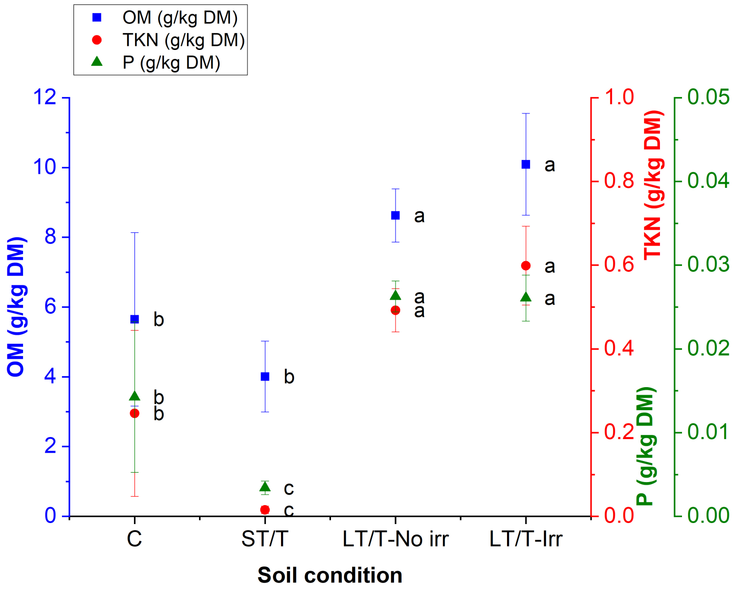
More specifically, the significant decrease in pH and increase in EC measured in the short term after irrigation with OMW (7.92 ± 0.39 and 594 ± 166 µS/cm, ST/T plots) restored in the long term in the treated soils (8.68 ± 0.09 and 157 ± 8.53 µS/cm in LT/T/No-irr, and 8.77 ± 0.06 and 221 ± 30.7 µS/cm in LT/T/Irr) to the values that were typical of the untreated soils (8.55 ± 0.43 and 267 ± 182 µS/cm, C plots).

Long-term irrigation resulted in significantly higher OM (10.1 ± 1.46 $g/kg_{DM}$, LT/T/Irr, and 8.63 ± 0.76 $g/kg_{DM}$, LT/T/No-irr), TKN (0.60 ± 0.09 $g/kg_{DM}$, LT/T/Irr, and 0.49 ± 0.05 $g/kg_{DM}$, LT/T/No-irr) and P (0.026 ± 0.003 $g/kg_{DM}$ for both LT/T/Irr and LT/T/No-irr) compared to the soils irrigated with OMW in the short term (4.01 ± 1.01 $g/kg_{DM}$ for OM, 0.02 ± 0.01 $g/kg_{DM}$ for TKN, and 0.003 ± 0.001 $g/kg_{DM}$ for P), and the untreated soils (5.65 ± 2.48 $g/kg_{DM}$ for OM, 0.25 ± 0.2 $g/kg_{DM}$ for TKN, and 0.014 ± 0.001

$g/kg_{DM}$ for P). The differences in soil OM, TKN and P between the latter two soil conditions were also significant.

Finally, the same patterns (significant increases in the long term, and decreases in the short term) compared to the control soils were noticed for K and Na. More specifically, LT/T/Irr soils and lT/T/No-irr plots showed a K of 0.17 ± 0.02 $g/kg_{DM}$ and of 0.16 ± 0.02 $g/kg_{DM}$, and a Na of 0.11 ± 0.02 $g/kg_{DM}$ and of 0.12 ± 0.01 $g/kg_{DM}$, respectively), while these cations decreased to 0.01 ± 0.01 $g/kg_{DM}$ in ST/T and 0.08 ± 0.06 $g/kg_{DM}$ for K and to 0.004 ± 0.001 $g/kg_{DM}$ in ST/T and 0.05 ± 0.046 $g/kg_{DM}$ for Na (Figure 4).


pH
EC
pH (-)
EC (µs/cm)
9.0
8.5
8.0
7.5
7.0
1000
800
600
400
200
0
b
b
c
c
a
a
a
a
C
ST/T
LT/T-No irr
LT/T-Irr
Soil condition


OM (g/kg DM)
TKN (g/kg DM)
P (g/kg DM)
OM (g/kg DM)
TKN (g/kg DM)
P (g/kg DM)
12
10
8
6
4
2
0
1.0
0.8
0.6
0.4
0.2
0.0
0.05
0.04
0.03
0.02
0.01
0.00
b
b
b
b
c
c
a
a
a
a
a
a
C
ST/T
LT/T-No irr
LT/T-Irr
Soil condition

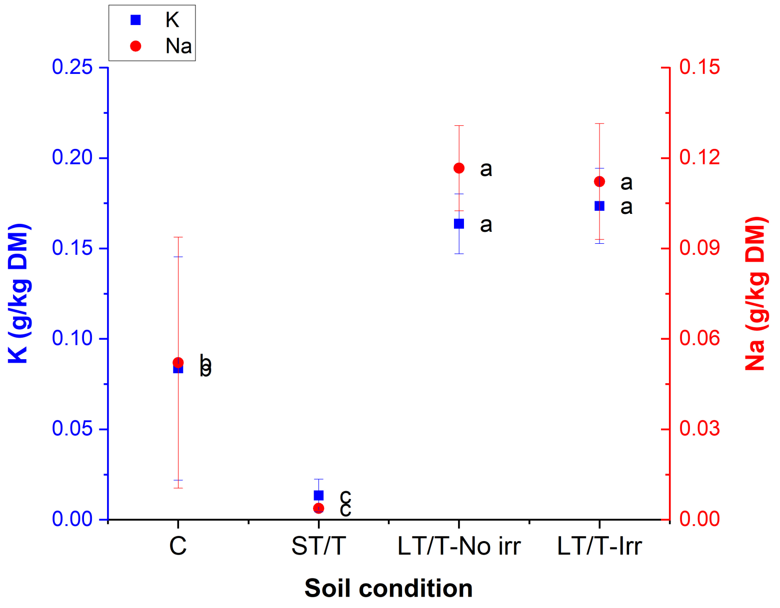

K
Na
K (g/kg DM)
Na (g/kg DM)
0.25
0.20
0.15
0.10
0.05
0.00
0.15
0.12
0.09
0.06
0.03
0.00
b
b
c
c
a
a
a
a
C
ST/T
LT/T-No irr
LT/T-Irr
Soil condition

Figure 4 - Mean ± standard deviation of soil properties measured under four soil conditions (control - C, treated in the short term, 2005-2007 – ST/T, long-term treated with OMW and non-irrigated in 2022 - LT/T-No irr, and long-term treated and irrigated with OMW in 2022 and 2023 – LT/T-Irr) in the experimental plots (Sfax, Tunisia).

*Legend: EC = electrical conductivity; SWC = Soil Water Capacity; CEC = Cation Exchange Capacity; OM = organic matter; TKN = Total Kjeldahl Nitrogen; PP = polyphenols. Different letters indicate significant differences after Tukey's test at* $p < 0.05$.

## 4 Discussion

Literature generally reports contrasting effects of OMW reuse for soil irrigation, which must be ascribed to large variability in wastewater characteristics of this effluent, in turn due to olive tree species and oil extraction method (Vaz et al., 2024) as well as application rates, climate, and soil type (Barbera et al., 2014; Shabir et al., 2023; Zema et al., 2019) (Table 5). Regarding soils, these effects may be the enhancement of soil fertility on one side, and phytotoxicity, groundwater contamination, compaction, salinisation, and acidification on the other side (Chatzistathis & Koutsos, 2017). In other words, nutrients and organic matter supplied to soil with OMW theoretically improve plant development and productivity when used properly. On the other side, OMW have also negative impacts on the environment, such as an enhanced risk of runoff of contaminants into surface waters or nutrient leaching into groundwater as well as increases in soil salinity and acidification, especially in dry areas, which can be detrimental to agricultural productivity and soil health.

### 4.1 Effects of long-term irrigation with OMW on soil properties

OMW application to the experimental olive grove resulted in significant changes in the monitored soil properties compared to the control. The most detrimental variations in the topsoil were detected in increases in EC (+66%), Cl (+63%) and PP (+122%) after annual irrigation, while intermittent irrigation was able to limit these increases for EC (+6.39%) and Cl (+10.2%), but not for PP, which were higher by 74.1% compared to the non-irrigated sites. In the deeper layer of soil with annual irrigation, the impacts on these properties were of lower magnitude compared to the topsoil, but always noticeable (+80.3% for Cl and +70.9 for PP). For the latter parameter, a high increase in the deeper layer was also noticed after intermittent irrigation (+96.4%). It is therefore evident the negative impacts on soil quality due to the increase in PP concentration, and this evidence is in close agreement with other studies, generally showing that the high content of phenolic compounds (over 50% of the original content in the processed olives) is one of the limiting factors for OMW application on soil, since a high content in PP is the main reason for its toxicity for plants and microorganisms (Barbera et al., 2013; Shabir et al., 2023; Vaz et al., 2024). Moreover, PPs are heavily biodegradable, and this leads to their long-term persistence in the treated soil with detrimental effects on soil microbiota (Fan et al., 2022) Also other authors report an increase in polyphenol content after application of OMW (Di Serio et al., 2008; Saadi et al., 2007; Sierra et al., 2007; Zenjari & Nejmeddine, 2001), but the phenol content of soil gradually decreases over time thanks to the degradation action of bacteria (Di Bene et al., 2013; Sierra et al., 2007; Vaz et al., 2024; Zema et al., 2019), which, however, depends on the environmental conditions (Barbera et al., 2013). Other authors, instead, observed noticeable concentrations of polyphenols several years after the last application of OMW (Feria, 2000; Kavvadias et al., 2014), especially in soils rich in carbonates and clay particles, where leaching is difficult and slow (Kavvadias et al., 2010, 2010; Sierra et al., 2007).

Table 5 – Summary of the main features and findings of studies dealing with OMW application on soils.

| **Author and year** | **Work country** | **OMW dose applied ($m^3$ $ha^{-1}$ $yr^{-1}$)** | **Observation period*** | **Soil variables monitored**** | **Main results** |
|---|---|---|---|---|---|
| Yamani et al., 2020 | Morocco | Not reported. | S | pH, EC, CEC, SWC, OM, PP, Cl, TKN | - Increase in indices of organic load<br>- Elevated PP and salt levels |
| Albalasmeh et al., 2019 | Jordan | 0, 50, 100, and 200 $m^3$ $ha^{-1}$ $year^{-1}$ | S | Aggregate stability, penetration resistance, hydraulic conductivity, water repellency | Degradation in soil's hydraulic and physical properties |
| Comegna et al., 2022 | Italy | Not reported. | S | Breakthrough curves, water retention curves, saturated hydraulic conductivity | Alteration in soil aggregates and porosity distribution, with a general rise in micropores and a decrease in macropores |
| Gargouri et al., 2022 | Tunisia | 50, 100 and 200 $m^3$ $ha^{-1}$ $year^{-1}$ | S | pH, EC, Ca, Mg, OM, P, K, Na, N | - Alterations in soil pH, EC, P, K and Ca levels in all soil layers<br>- Decreases in Mg, Na and OM with treated OMW<br>- Decreases in K, Ca, Mg, and Na with crude OMW |

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| | | | | | - Improvements in N |
| Mohawesh et al., 2019 | Jordan | 20, 40, 60, 80, and 120 $m^3$ $ha^{-1}$ $year^{-1}$ | S | Biological yield, grain yield, harvest index | - At all OMW application doses, no adverse effects on soil parameters and wheat growth.<br>- A 60 $m^3$ $ha^{-1}$ OMW application rate, high increases in wheat growth without any substantial detrimental effect on soil quality |
| Chaâri et al., 2022 | Tunisia | 40 and 80 $m^3$ $ha^{-1}$ $year^{-1}$ | S | EC, Cl, Na, OM, P, N, K, germination test | - Higher levels of OM, phosphate, N, and K for the two doses<br>- Clay type’s influence on the phenolic compounds' ability to bind to soil particles<br>- Beneficial effects on germination index of tomato and alfalfa seeds<br>- Temporary decreases in EC, Na, and Cl |
| Zema et al., 2019 | Italy | 80 $m^3$ $ha^{-1}$, $year^{-1}$ | S | pH, EC, CEC, SWC, OM, TKN, P, ions, PP, VolAcid, ASIndex | - Increases in OM, potassium, and phosphorus and increased the stability of the aggregates<br>- Lower levels of minerals, OM, and PP in the deeper soil layer<br>- Increases in K, Ca and Na sodium in both the topsoil and deeper layers |
| Di Serio et | Italy | 8 and 16 L | S | pH, EC, OM, TKN, PP,K | - Rise in phenols and OM |

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| al., 2008 | | m$^{-2}$ | | | - Lower N content<br>- Increase (+20–30%) in soil C/N. |
| Piotrowska et al., 2011 | Italy | 80 m$^{3}$ ha$^{-1}$ year$^{-1}$ | S | pH, EC, CEC, OM, TKN, C/N, P, ions, PP, enzymes | - Different paths in tested enzymes as well as the microbial and chemical soil properties in response to both crude and dephenolized OMW addition and time of incubation.<br>- Sudden changes (0–14 days after the addition of OMW) in several properties of the soil, mostly biological<br>- Quick return to their original values, demonstrating the soil's resilience capability |
| Sierra et al., 2007 | Spain | 30, 180, 360 m$^{3}$ ha$^{-1}$ year$^{-1}$ | S | pH, EC, OM, TKN, P, ions, PP | - Increases in P, OM and N at higher rates of OMW application<br>- Temporary N immobilisation, higher salinity levels and higher concentrations of phenolic compounds (however, quickly biodegraded) |
| Saadi et al, 2007 | Israel | 36 and 72 m$^{3}$ ha$^{-1}$ year$^{-1}$ | S | Respiration, hydrolytic activity, C, pH, PP | - Increases in microbial counts, soil respiration and hydrolytic activity<br>d. following the high OMW application level<br>- Short-term effect on soil phytotoxicity |

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|  |  |  |  |  | - Lack of a negative effect on soil activity |
| Kavvadias et al., 2014 | Greece | Not reported. | L | pH, EC, K, Mg, Cl, $SO_2$, OM, PP, N, P, B, Cu, Fe, Mn, Zn | - Significant increases in OM, PP, TN and inorganic N, accessible micronutrients, EC, P, K, and Mg, mostly in surface soil layers.<br>- In the long term (8 years), residual quantities of available P, exchangeable K, total and inorganic N, PP, Fe, and Cu |
| Kavvadias et al., 2010 | Greece | Not reported. | S | pH, EC, K, Mg, Cl, $SO_4^{2+}$, OM, PP, N, P, $CaCO_3$, mineral elements | - Significant increases in soil PP, TN, available P, EC, Cl, $SO_4^{2-}$, $PO_4^{3-}$, $NH_4^+$, Cu, Mn, Zn, Fe, available B and especially $K^+$<br>- Decrease in carbonate content<br>- Long-term, uncontrolled raw OMW application on soils may modify soil characteristics and raise the possibility of groundwater pollution. |
| Di Bene et al., 2013 | Italy | 80 $m^3$ $ha^{-1}$ $year^{-1}$ | S+L | pH, EC, K, N, microbial biomass carbon, soil respiration, root colonisation, arbuscules, vesicles | - Effects on primary soil chemical and biological parameters in the short term, but unable to ascertain long-term residual impacts<br>- Reduced AM fungal root colonization following OMW applications |

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| | | | | | - enhanced arbuscule occurrence throughout both disposal times, both in the short- and long-term |
| Vella et al., 2016 | Italy | 30 $m^3$ $ha^{-1}$ $year^{-1}$ | S | OM, pH, EC, SWC, N, Ca, Na, Mg, K, metals, PP | - No significant changes in soil OM and nutrient, despite long-lasting and repeated OMW applications<br>- minor effects on pH, EC, OM, key cations, and PP |
| Belaqziz et al., 2016 | Morocco | 10 L $m^{-2}$ $year^{-1}$ | S | pH, EC, Na, K, Ca, N, P, PP | - A sudden enrichment in mineral and nutritive elements decreased three months after OMW application with biodegradation in calcareous soil<br>- At high doses, important increase in soil physicochemical characteristics (EC, P, N, OM, and soluble PP<br>- Increase in soluble PP in the upper layer of soil |
| Wang et al., 2007 | China | Not reported. | S | Heavy metals, specific retention, void fraction, bulk density, hydraulic | - No plant diseases and discernible effects on loess soil<br>- Slight increase in OM |

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| | | | | conductivity, pH, salinity, OM | |
| Shahalam et al., 1997 | Jordan | Not reported. | S | pH, EC, OM, TKN, P, ions | - Non-significant differences in alfalfa and radish plants for B, Pb and Cd between the freshwater and wastewater irrigation<br>- Slight changes in soil porosity and salinity with wastewater irrigation<br>- Slight and non-significant changes in pH |
| El Hassani et al., 2020 | Morocco | 500 g of raw or sterilized soil + 200 ml of raw or sterilized OMW | L | PP, humic acid | -Non-significant difference in PP between treated soil and control<br>- Humic acids in the presence of OMW with proteins, carbonated compounds, PP and long-chain aliphatics<br>-Persistence of PP in soil humus |
| Ayoub et al., 2014 | Jordan | 0.5, 10, 20 L $m^{-2}$ at one dose, and 20 L $m^{-2}$ at four equal doses | S | pH, EC, SWC, OM, TKN, P, ions, PP , metals | - No detrimental effects on soil properties<br>- Significantly higher K, OM, PP, and total microbial<br>- Suggested application of OMW at an annual rate of 10 L $m^{-2}$ |
| Dakhli et al., | Tunisia | 15 to 45 $m^3$ | S | pH, EC, ions, P, K, C,N, PP | - Significant impact on soil chemical and |

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| 2020 | | ha$^{-1}$ year$^{-1}$ | | | microbiological properties<br>- The dose of 15 m$^3$ ha$^{-1}$ is appropriate for the vegetative development of the *Faba* bean<br>- Stable pH and, but increase in soil salinity at the highest dose |
| Montemurro et al., 2011 | Italy | 80 and 120 m$^3$ ha$^{-1}$ year$^{-1}$ | S | C, N, P, K, humic and fulvic acids | - Improvement in OM in soil treated with OMW<br>- Increases in available K and P between treated and control soils at both doses |
| Chaari et al, 2015 | Tunisia | 50, 100 and 200 m$^3$ ha$^{-1}$ year$^{-1}$ | L | pH, EC, OM, P, N, ions, PP | - Increase in topsoil EC, OM, K, P, N<br>- Decrease in Ca$^{2+}$<br>- No pH alterations<br>- Alterations in exchangeable sodium percentage and soil sodium adsorption ratio.<br>- High levels of PP in the upper soil layers with fluctuations not being proportional to the OMW dose |
| Mekersi et al., 2022 | Algeria | 50–80 t ha$^{-1}$ year$^{-1}$ | S | pH, EC, OM, P, C, N, PP | - Increase in PP, OM, OC, and EC<br>- High concentration of PP<br>- No discernible effects on P or TN |

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| Magdich et al., 2013 | Tunisia | 50, 100, and 200 m$^3$ ha$^{-1}$ year$^{-1}$ | S | pH, EC, OM, P, K, N, ions | - At higher OMW concentration and application frequency drop in pH and increases in EC, OM, TN, Na, and K<br>- Slow increases Ca and Mg<br>- No changes in K |
| Khalil et al., 2024 | Syria | 0, 5, 10 and 15 L m$^{-2}$ year$^{-1}$ | S | pH, EC, OM, SWC, metals, N, ions, P, K,CaCO$_3$ | - Improvements in OM, macro- and micronutrient (proportional to OMW applied)<br>- Nutrient imbalance |
| Aharonov-Nadborny et al., 2017 | Israel | Not reported. | S | pH, EC, metals, ions, CaCO$_3$ | - pH buffering, cation exchange capacity, and clay had a role in the mobilization of native soil metals<br>- Significant metal leaching<br>- Combination between OM with metal cations generating precipitating or moving complexes |
| Moraetis et al., 2011 | Greece | Not reported. | S | pH, EC, CEC, metals, ions | - High increases in N and K but decrease in P<br>- Enhanced microbial activity due to breakdown of organic N and C<br>- No evident increase in heavy metal deposition in soil |

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| | | | | | - Stable EC (below the salinisation threshold)<br>- No effects on the quality of groundwater |
| Tamimi et al., 2016 | Palestine | Not reported. | S+L | pH, EC, SWC, temperature, water drop penetration time, PP | - Leaching of OMW components to groundwater during the rainy season and by formation of preferential flow paths before the rain season<br>- Persistence of negative effects in long hot and dry periods due to accumulation and polymerization of OMW |
| Mahmoud et al., 2010 | Syria | Not reported. | L | Water drop penetration time, hydraulic conductivity, infiltration rate | - Under increasing OM content, enhanced soil hydrophobicity and lowered drainable porosity<br>- Decrease in soil's hydraulic conductivity, but increase in infiltration rate due to large and deep shrinkage cracks 15 years after treatment |
| Melgar et al., 2009 | Spain | Not reported. | L | EC, ions | - No salinity effects after 8 years of treatment<br>- Low leaf $Na^+$ and $Cl^-$ concentrations (below the toxicity thresholds for plants)<br>- No salt accumulation due to leaching by rainwater |

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| Kurtz et al., 2021 | Germany | 50, 100, 100 with tillage and 150 m$^{3}$ ha$^{-1}$ year$^{-1}$ | S | SWC, specific UV absorbance, water drop penetration time, pH, EC, C, P | - Increase in SWC, several biological and organic matter indices, total PP and ions<br>- S hydrophobicity in the topsoil .<br>- OMW leaching hazard at doses of 50 and 100 m$^{3}$ ha$^{-1}$ year$^{-1}$ applied every two years followed by tillage |
| This study | Tunisia | 50 m$^{3}$ ha$^{-1}$ year$^{-1}$ | L + S | pH, EC, CEC, SWC, OM, TKN, C/N, P, ions, PP | - Increased EC, PP, OM, TN, and P under both intermittent and annual irrigation with OMW<br>- Most pronounced changes in the topsoil, with intermittent irrigation showing a moderate impact on EC and Cl levels compared to annual irrigation<br>- Noticeably increased PP and soil salinity |

Note: * L = long-term (> 10 years), S = short-term (< 5 years); EC = electrical conductivity; CEC = cation exchange capacity; SWC = soil water content; OM = organic matter; TKN = total Kjehldal Nitrogen; PP = polipheno.

The increase in soil EC also requires attention when OMW must be applied to soil, since it indicates a possible salt accumulation in soil. This effect of OMW may result in the degradation of the soil structure with a consequent decrease in the soil hydraulic conductivity (Chatzistathis & Koutsos, 2017; Zema et al., 2019) as well as other in adverse effects on plants (Zema et al., 2019). This impact is detrimental also because an increase in soil EC result in a reduction in crop productivity due to the increase in soil osmotic potential and therefore stress for plants in uptaking water and nutrients (Butcher et al., 2016; Zema et al., 2019). In this regard, it is worth noting that, in our study, the increase in Na, which is often the major responsible for soil structure degradation among ions, is moderate (peak of +40.9% after intermittent irrigation in the deeper layer), and an accumulation of this salt is evident also in the surface layer of soil (+25-30% for both irrigation strategies). A proper control of OMW application rates is suggested to mitigate this undesired impact, and therefore the soil hydraulic conductivity may be considered a key parameter for determining the volume of OMW that can be spread on soil (Barbera et al., 2013), since the latter is proportional to the salt amount that is stored into the treated soil (Mekki et al., 2009; Sierra et al., 2007). However, the variability in EC in soil treated with OMW depends on several factors, such as the olive oil extraction system (Barbera et al., 2013; Roig et al., 2006). The leaching action of rainfall generally reduces the soil EC and PP in soils and potentially transfers salts to the groundwater (Barbera et al., 2014; Sierra et al., 2007), thus reducing the impact of OMW application on soil (Vella et al., 2016). However, the rainfall input in the studied area is low and the leaching effects are limited, which indicates that salt accumulation in the treated soils is persistent over time.

The increase in chlorine content in the treated soils must also be a threat of this practice, since this element could be toxic for some crops (Gori et al., 2000; Karaivazoglou et al., 2005; Pedrero & Alarcón, 2009). Plants can uptake and store chlorine, chlorides and chlorinated compounds, and this can be adverse for their growth and development of microbial communities together with sodium (Gori et al., 2000). Chlorine and chlorinated compounds may also alter some chemical and biochemical properties of soils (Pedrero et al., 2012; Shahalam et al., 2007; Wang et al., 2007). Therefore, the Cl content of soil must be carefully monitored (especially in the dry areas as the studied sites), if the clear water used for olive oil extraction in mills is noticeably higher compared to the crop tolerance (Zema et al., 2023).

Conversely, the fertilising effects of OMW were noticeable and positive for both irrigation strategies, since OM, N, P and K were noticeably higher at both soil depths compared to the control (peaks of +56.6% of TKN, 43.1% of OM and 29.1% of P in the topsoil). In general, and as expected, these increases were more noticeable in the topsoil compared to the deeper layers. A higher CEC (+15 ± 20%) is noticed for both irrigation strategies in both soil layers. Several studies observed general improvements in soil fertility, due to storage of OM and nutrients. These effects are highly beneficial in semi-arid soils, which are generally characterized by low organic content, phosphorus, potassium, and nitrogen, since the use chemical fertilizers can be drastically reduced (Belaqziz et al., 2016; Killi & Kavdır, 2013; Shabir et al., 2023). However, about OM fate in soils treated with OMW, the literature is contrasting, since almost all studies observed increases in organic matter or carbon content of the topsoil, especially in the long term, due to the high organic matter content of the effluent (Ayoub et al., 2014; Ben Rouina et al., 2006; Chatzistathis & Koutsos, 2017), while this accumulation was not observed by few authors (Di Bene et al., 2013; Sierra et al., 2007; Vaz et al., 2024). This effect may lead to a general improvement in important physical properties of soil, such as the increase in water retention capacity and infiltrability. The enhancement in OM and nutrient content in the treated soils under the dry conditions of the study area should exclude some expected detrimental impacts, such as the possibility of groundwater pollution due to the percolation of organic compounds and compounds, given the low rainfalls in these semi-arid areas. Regarding the dynamics of nutrients after OMW application, our study is in close agreement with the literature, which is quite consistent in stating increases in nitrogen, phosphorus and potassium concentrations (e.g., Ayoub et al., 2014; Belaqziz et al., 2016; Chartzoulakis et al., 2006; Chatzistathis & Koutsos, 2017) - although being variable with soil depth (El Hassani et al., 2020; Vaz et al., 2024) - with few exceptions. No changes or reductions in nutrients of soils irrigated with OMW were observed, for instance, by Chaari et al. (2015) for phosphorus and potassium, Mechri et al. (2011) and Montemurro et al., (2011) for potassium and phosphorus, Sierra et al. (2007) for phosphorus and nitrogen, and (Piotrowska et al., 2011) for nitrogen.

Despite the different variations in total organic carbon and nitrogen (on average close to 10), this study did not show a noticeable unbalance in their ratio in the treated soils compared to the control, as shown by the maximum difference measured in the topsoil irrigated with OMW (+8.51%). This unbalance, which is expected because OMW is of vegetal origin and therefore poor in nitrogen, could be another constraint of the OMW application on soil, the organic N content of OMW being usually low, whereas the organic matter concentration being high (approximately 65% of the OMW dry weight, Barbera et al., 2013). Increases in the C/N ratio up to 50 were reported by Di Serio et al., 2008, and this can reduce the mineralization rate of organic compounds and immobilise inorganic nitrogen, N being required by bacteria (Barbera et al., 2013; Mekki et al., 2009).

The values of pH were slightly, but always significantly, modified by the OMW application, while the increase in water storage was practically negligible (less than +2%). Therefore, the expected reduction in soil pH, due to the acidity of the used OMW, was not observed, and this may have happened for thre reasons: first, the buffering capacity is presumably sufficient to balance significant changes in pH (Ayoub et al., 2014; Vaz et al., 2024; Zema et al., 2019); second, the very quick recovery of pH to normal levels (Chatzistathis & Koutsos, 2017; López-Piñeiro et al., 2011; Magdich et al., 2013); and third, the low annual precipitation, leading to very low leaching of soil acids. This non-negative impact of OMW application on soil is beneficial for soil health, since pH regulates the availability of nutrients for microorganisms and plants (El Hassani et al., 2023; Vaz 2024). Soil acidification due to application of effluents with high acidity is a detrimental effect for this practice, since alterations in soil pH lead to a decrease in crop productivity or soil alkalinisation with consequent structure degradation (Steinmetz et al., 2015; Vaz et al., 2024). Also, the effects of irrigation with OMW on soil pH are contradictory, some studies reporting reductions in the short and long term after irrigation (e.g., Chaari et al., 2015; Di Serio et al., 2008; Sierra et al., 2007; Zema et al., 2019), but others, although few, recording even slight increases (Chartzoulakis et al., 2010; Di Bene et al., 2013; Piotrowska et al., 2011). Overall, according to the significant changes in the monitored soil properties, the sites treated with intermittent irrigation seem to be more similar to the untreated soils compared to the sites subject to annual OMW applications, as revealed by the AHCA. This suggests reducing the frequency of OMW applications to soils to inter-annual irrigations, in order to limit the possible detrimental impacts to the olive groves.

### 4.2 Effects of short-term and long-term irrigation with OMW on soil properties

A few studies have evaluated the long-term effect of OMW application on soil properties (Chaari et al., 2015; Kavvadias et al., 2014; Vella et al., 2016). According to this insufficient body of literature, this study has contributed to investigating whether the long-term effects of this practice are in line with the short-term impacts.

Compared to the control, the short-term decrease in pH (-7.5%) due to OMW application is counteracted many years after the treatment starts, when pH is stable or slightly decreased. Also, the sudden increase in EC (+123%) becomes moderate in the long term, although noticeable (+20 ± 40%). What requires much caution is the stable increase in soil Na, which becomes two-fold the value measured in the short term (+92.7%) and persists significantly and alarming several years after the irrigation starts (+110 ± 120%). Also Dakhli et al. (2021) measured increases in salinity after some years from the start of irrigation with OMW.

The fertilisation effects of OMW application are strengthened over time. In more detail, the short-term increases in TKN (+93.7%), P (76.1%) and K (84%) slightly increase with intermittent irrigation (+100%). The increase in the latter two parameters is stable (+80 ± 85% for P and +100 ± 110% for K) also with annual application, which instead increases soil nitrogen (+140%) more than compared to the other irrigation strategy (+100%). Long-term soil treatment with OMW results in higher increases in OM content of soil compared to the short-term (+29%), and more in the case of annual irrigation (+80%) compared to intermitted application (+50%). Long-term increases in organic matter and nutrients after OMW application are also reported by Chaari et al., (2015), Chartzoulakis et al. (2010) and Mekki et al. (2007). According to Vella et al. (2016), the application of OMW on soil, even if repeated for many years, plays non-significant impacts on pH, OM, concentrations of main cations and PP content on the treated sites.

## 5 Conclusions

In reply to the first research question, this study has demonstrated that, compared to the untreated sites, all studied physico-chemical properties of soils treated with OMW significantly changed, regardless of the irrigation strategy (intermittent or annual application of the effluents). Noticeable increases were measured for soil salinity and content in polyphenols, which led to paying attention to these aspects of soil quality. Intermittent irrigation or irrigation with clear water after application of OMW may be a feasible strategy to leach salts and polyphenols in soils (limited due to the low rainfalls in the studied semi-arid area), to limit salinisation and possible toxicity for plants and microorganisms. Conversely, the fertilisation effects in treated soils thanks to the application of the organic matter and nutrients in OMW are beneficial and long-lasting (as shown by the comparison to the short-term effects, evaluated by a parent study). This positive impact of OMW land spreading allows for saving fertilisers and water, the latter being an essential added value of irrigation with OMW under the expected climate change scenarios . No noticeable changes in soil pH were also detected, and this contrasts the expected risk of soil acidification due to the application of acidic effluents. The study has also shown that, in the long term, some undesired effects of OMW application detected on soils a few years after irrigation starts reduced, and this replies to the second research question. The sudden increase in soil pH and salinity becomes very low or moderate, respectively, but attention must be paid to the sodium content of soil, to avoid salinisation and degradation of its structure. Annual or inter-annual applications of OMW make stable or even increase the level of soil fertility measured in the short term. However, the continuous application of OMW to soil is less advisable compared to the intermittent ferti-irrigation, this wastewater being rich in nutrients, salts and acids, often in excess compared to soil buffering capacity. This may lead to undesired effects, and even damage the environment (e.g., fertilizer leakage into groundwater or runoff into surrounding water bodies in the case of extreme rainfalls) or crops (risks of soil salinisation that negatively impacts on plant root development and water uptake).

Possible limitations of this study may be: (i) the lack of attention to some key properties of soil under the analysed treatments, which require more investigations (for instance, exploring how the enzymatic activities and microbial communities respond to the OMW application to soil, considering the close relationships between the enzyme and microbial characteristics and physico-chemical properties of soil after OMW land spreading) and (ii) only one study area, which makes the results quite site-specific, while experiments in other environments with similar or different climatic and pedological characteristics may be welcome for a broad validation of the results.

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## Author Contributions

Conceptualization: S.A., M.C. and A.M.; methodology, data collection and original data analysis: S.A., and A.M.; data presentation, writing: S.A, D.A.Z., M.E.L.B. and A.M.; reviewing and editing: S.A, D.A.Z., M.E.L.B. and A.M.; funding acquisition: M.C. and A.M. All authors have read and agreed to the published version of the manuscript.

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## DECLARATIONS

### Data Availability

All relevant data are included in this document.

### Ethics Approval and Consent to Participate

Not applicable

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### Competing Interests

The authors declare no competing interests.

## References

Abdennbi, S., Chaieb, M., & Mekki, A. (2023). Long-term effects of olive mill waste waters spreading on the soil rhizospheric properties of olive trees grown under Mediterranean arid climate. *Soil Research*, 62. https://doi.org/10.1071/SR23102

Albalasmeh, A.A., Alajlouni, M.A., Ghariabeh, M.A., & Rusan, M.J. (2019). Short-term effects of olive mill wastewater land spreading on soil physical and hydraulic properties. *Water, Air, & Soil Pollution*, *230*, 1–14.

Aloui, F., Abid, N., Roussos, S., & Sayadi, S. (2007). Decolorization of semisolid olive residues of "alperujo" during the solid-state fermentation by Phanerochaete chrysosporium, Trametes versicolor, Pycnoporus cinnabarinus and Aspergillus niger. *Biochemical Engineering Journal, 35*, 120–125.

Aharonov-Nadborny, R., Tsechansky, L., Raviv, M., & Graber, E. R. (2017). Impact of spreading olive mill wastewater on agricultural soils: Leaching of metal micronutrients and cations. *Chemosphere, 179*, 213-221.

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. *Scientia Horticultura. 175*, 160–166.

Barbera, A.C., Maucieri, C., Cavallaro, V., Ioppolo, A., & Spagna, G. (2013). Effects of spreading olive mill wastewater on soil properties and crops, a review. *Agricultural Water Management, 119*, 43–53.

Barbera, A.C., Maucieri, C., Ioppolo, A., Milani, M., & Cavallaro, V. (2014). Effects of olive mill wastewater physico-chemical treatments on polyphenol abatement and Italian ryegrass (Lolium multiflorum Lam.) germinability. *Water Research, 52*, 275–281.

Belaqziz, M., El-Abbassi, A., Agrafioti, E., & Galanakis, C.M. (2016). Agronomic application of olive mill wastewater: Effects on maize production and soil properties. *Journal of Environmental Management, 171,* 158–165.

Ben Mbarek, H., Gargouri, K., Mbadra, C., Ben Mahmoud, I., Chaker, R., Maktouf, S., & Rigane, H. (2020). Effects of combination of tillage with olive mill wastewater on soil organic carbon groups in arid soils. *Arab Journal of Geosciences. 13,* 1-10. https://doi.org/10.1007/s12517-020-5235-x

Ben Rouina, B., Gargouri, K., Abichou, M., & Taamallah, H. (2006). Mill wastewater as an ecological fertilizer for olive tree orchards, in: Second International Seminar:"Biotechnology and Quality of Olive Tree Products Around the Mediterranenan Basin", Olivebioteq, Proceedings. pp. 139–141.

Bombino, G., Andiloro, S., Folino, A., Lucas-Borja, M.E., & Zema, D.A. (2021). Short-term effects of olive oil mill wastewater application on soil water repellency. Agricul Wat Manag. 244, 106563. https://doi.org/10.1016/j.agwat.2020.106563

Butcher, K., Wick, A. F., DeSutter, T., Chatterjee, A., & Harmon, J. (2016). Soil salinity: A threat to global food security. *Agronomy Journal*, 108(6), 2189-2200.

Calabrò, P.S., Fòlino, A., Tamburino, V., Zappia, G., & Zema, D.A., (2018). Increasing the tolerance to polyphenols of the anaerobic digestion of olive wastewater through microbial adaptation. *Biosystems Engineering, 172,* 19–28. https://doi.org/10.1016/j.biosystemseng.2018.05.010

Caon, L., Vallejo, V.R., Ritsema, C.J., & Geissen, V. (2014). Effects of wildfire on soil nutrients in Mediterranean ecosystems. *Earth-Science Review, 139,* 47–58.

Chaâri, L., Alsaiari, N. S., Amari, A., Ben Rebah, F., Kallel, M., & Mechichi, T. (2022). Soil responses to high olive mill wastewater spreading. *Agronomy, 12*(4), 972. https://doi.org/10.3390/agronomy12040972

Chaari, L., Elloumi, N., Mseddi, S., Gargouri, K., Rouina, B. B., Mechichi, T., & Kallel, M. (2015). Changes in soil macronutrients after a long-term application of olive mill wastewater. *Journal of Agriculture Chemistry and Environment*, *4*(01), 1. http://dx.doi.org/10.4236/jacen.2015.41001

Chartzoulakis, K., Psarras, G., Moutsopoulou, M., & Stefanoudaki, E. (2010). Application of olive mill wastewater to a Cretan olive orchard: Effects on soil properties, plant performance and the environment. *Agriculture, Ecosystems & Environment, 138,* 293–298.

Chartzoulakis, K., Psarras, G., Moutsopoulou, M., & Stefanoudaki, E. (2006). Application of olive mill wastewater on olive orchards: effects on soil properties, plant performance and the environment. *Proceedings of Olivebioteq, 2,* 37–44.

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. *Agricultural Water Management, 190,* 55–64.

Chiavola, A., Farabegoli, G., & Antonetti, F. (2014). Biological treatment of olive mill wastewater in a sequencing batch reactor. *Biochemical Engineering Journal, 85*, 71-78.

Comegna, A., Dragonetti, G., Kodesova, R., & Coppola, A. (2022). Impact of olive mill wastewater (OMW) on the soil hydraulic and solute transport properties. *International Journal of Environmental Science and Technology, 19,* 7079–7092.

Dakhli, R., Gosh, A., Wali, A., Chandra, M.M., & Khatteli, H. (2021). Agricultural valorization of olive mill wastewater in arid regions of tunisia: short-term impact on soil biochemical properties and faba bean growth. *Polish Journal of Environmental Studies, 30,* 1117–1128.

Dbara, S., Ouni, A., Brahim, M., & Fezai, N. (2019). Does olive mill wastewater stimulate olive plant growth? *Journal of Plant Nutrition, 42*(1), 58-66.

Di Bene, C., Pellegrino, E., Debolini, M., Silvestri, N., & Bonari, E. (2013). Short-and long-term effects of olive mill wastewater land spreading on soil chemical and biological properties. *Soil Biology and Biochemistry, 56,* 21–30.

Di Serio, M.G., Lanza, B., Mucciarella, M.R., Russi, F., Iannucci, E., Marfisi, P. & Madeo, A. (2008). Effects of olive mill wastewater spreading on the physico-chemical and microbiological characteristics of soil. *International Biodeterioration & Biodegradation, 62,* 403–407.

Dourou, M., Kancelista, A., Juszczyk, P., Sarris, D., Bellou, S., Triantaphyllidou, I.-E., Rywinska, A., Papanikolaou, S., & Aggelis, G. (2016). Bioconversion of olive mill wastewater into high-added value products. *Journal of Cleaner Production, 139,* 957–969.

El Hassani, F.Z., Fadile, A., Faouzi, M., Zinedine, A., Merzouki, M., & Benlemlih, M. (2020). The long term effect of Olive Mill Wastewater (OMW) on organic matter humification in a semi-arid soil. Heliyon 6.

El Kafz, G., Cherkaoui, E., Khamar, M., Benradi, F., & Nounah, A. (2023). Characterization of two olive mill wastewater and its effect on Fenugreek (Trigonella foenum-graecum) germination and seedling growth. *Journal of Ecological Engineering, 24*(11). https://doi.org/10.12911/22998993/171545

Es Saouini, H., Bouzid, S., Trankil, A., Amharref, M., & Bernoussi, A. S. (2023). Application of statistical methods for the comparative study of the degree of pollution of wastewater collected from three olive mills in Tangier-Tetouan-Al Hoceima region (Northern Morocco). *Journal of Ecological Engineering, 24*(4), https://doi.org/10.12911/22998993/160684.

Fader, M., Shi, S., von Bloh, W., Bondeau, A., & Cramer, W. (2016). Mediterranean irrigation under climate change: more efficient irrigation needed to compensate for increases in irrigation water requirements. *Hydrology and Earth System Science, 20,* 953–973.

Fan, D., Zhao, Z., Wang, Y., Ma, J., & Wang, X. (2022). Crop-type-driven changes in polyphenols regulate soil nutrient availability and soil microbiota. *Frontiers in Microbiology,* 13, 964039.

Feria, A.L. (2000). The generated situation by the OMW in Andalusia, in: Actas/Proceedings-Workshop Improlive-2000-Anexo A. pp. 55–63.

Foti, P., Romeo, F.V., Russo, N., Pino, A., Vaccalluzzo, A., Caggia, C., & Randazzo, C.L. (2021). Olive mill wastewater as renewable raw materials to generate high added-value ingredients for agro-food industries. *Applied Science, 11,* 7511.

Gargouri, B., Ben Brahim, S., Marrakchi, F., Ben Rouina, B., Kujawski, W., & Bouaziz, M. (2022). Impact of wastewater spreading on properties of Tunisian soil under arid climate. *Sustainability, 14,* 3177.

Gori, R., Ferrini, F., Nicese, F.P., & Lubello, C. (2000). Effect of Reclaimed Wastewater on the Growth and Nutrient Content of Three Landscape Shrubs. *Journal of Environmental Horticulture, 18,* 108–114. https://doi.org/10.24266/0738-2898-18.2.108

Hamimed, S., Landoulsi, A., & Chatti, A. (2021). The bright side of olive mill wastewater: valuables bioproducts after bioremediation. *International Journal of Environmental Science and Technology,* 1-22. https://doi.org/10.1007/s13762-021-03145-0

Hodaifa, G., Gallardo, P. A. R., García, C. A., Kowalska, M., & Seyedsalehi, M. (2019). Chemical oxidation methods for treatment of real industrial olive oil mill wastewater. *Journal of the Taiwan Institute of Chemical Engineers, 97*, 247-254.

Jamrah, A., Al-Zghoul, T.M., & Darwish, M.M. (2023). A comprehensive review of combined processes for olive mill wastewater treatments. *Case Studies in Chemical and Environmental Engineering,* 100493.

Kandeler, E., & Gerber, H. (1988). Short-term assay of soil urease activity using colorimetric determination of ammonium. *Biology and fertility of Soils, 6,* 68–72.

Karaivazoglou, N.A., Papakosta, D.K., & Divanidis, S. (2005). Effect of chloride in irrigation water and form of nitrogen fertilizer on Virginia (flue-cured) tobacco. *Field Crops Research, 92,* 61–74. https://doi.org/10.1016/j.fcr.2004.09.006

Kavvadias, V., Doula, M., & Theocharopoulos, S. (2014). Long-term effects on soil of the disposal of olive mill waste waters (OMW). *Environment Frenzies, 15,* 37–51.

Kavvadias, V., Doula, M.K., Komnitsas, K., & Liakopoulou, N. (2010). Disposal of olive oil mill wastes in evaporation ponds: effects on soil properties. *Journal of Hazardous Materials, 182,* 144–155.

Khalil, J., Habib, H., Bouguerra, S., Nogueira, V., & Rodríguez-Seijo, A. (2024). The impact of olive mill wastewater on soil properties, nutrient, and heavy metal availability: A study case from Syrian vertisols. *Journal of Environmental Management, 351*, 119861.

Killi, D., & Kavdır, Y. (2013). Effects of olive solid waste and olive solid waste compost application on soil properties and growth of Solanum lycopersicum. *International Biodeterioration & Biodegradation,* 82, 157–165.

Klute, A., 1986. Methods of soil analysis, Part 1. Agronomy 9.

Knechtel, J.R. (1978). A more economical method for the determination of chemical oxygen demand. *Water and Pollution Control, 166,* 25–29.

Kottek, M., Grieser, J., Beck, C., Rudolf, B., & Rubel, F. (2006). World map of the Köppen-Geiger climate classification updated.

Kurtz, M. P., Dag, A., Zipori, I., Laor, Y., Buchmann, C., Saadi, I., ... & Diehl, D. (2021). Balancing the pros and cons of spreading olive mill wastewater in irrigated olive orchards. *Processes, 9*(5), 780.

López-Piñeiro, A., Albarrán, A., Nunes, J.R., Peña, D., & Cabrera, D. (2011). Cumulative and Residual Effects of Two-Phase Olive Mill Waste on Olive Grove Production and Soil Properties. *Soil Science Society of America Journal, 75,* 1061–1069.

Lu, Y., Song, S., Wang, R., Liu, Z., Meng, J., Sweetman, A. J., ... & Wang, T. (2015). Impacts of soil and water pollution on food safety and health risks in China. *Environment International, 77*, 5-15.

Magdich, S., Ahmed, C. B., Jarboui, R., Rouina, B. B., Boukhris, M., & Ammar, E. (2013). Dose and frequency dependent effects of olive mill wastewater treatment on the chemical and microbial properties of soil. *Chemosphere 93*(9), 1896-1903. https://doi.org/10.1016/j.chemosphere.2013.06.066.

Mahmoud, M., Janssen, M., Haboub, N., Nassour, A., & Lennartz, B. (2010). The impact of olive mill wastewater application on flow and transport properties in soils. *Soil and Tillage Research, 107*(1), 36-41.

Mahmoud, I.B., Mbarek, H.B., Gargouri, K., Rigane, H., Medhioub, M., & Medhioub, K. (2022). Effects of long-term olive mill wastewater disposal on the physical properties of an olive orchard soil in an arid Mediterranean region. *Arab Journal Geoscience, 15,* 519. https://doi.org/10.1007/s12517-022-09644-9.

Mechri, B., Cheheb, H., Boussadia, O., Attia, F., Mariem, F.B., Braham, M., & Hammami, M. (2011). Effects of agronomic application of olive mill wastewater in a field of olive trees on carbohydrate profiles, chlorophyll a fluorescence and mineral nutrient content. *Environmental and Experimental Botany, 71,* 184–191.

Mekersi, N., Addad, D., Kadi, K., Casini, S., Hackenberger, D. K., Boumaza, A., & Lekmine, S. (2023). Effects of olive mill wastewater and olive mill pomace on soil physicochemical properties and soil polyphenols. *Journal of Materials Cycles and Waste Management, 25(3),* 1404-1416. https://doi.org/10.1007/s10163-023-01618-7

Mekersi, N., Kadi, K., Casini, S., Addad, D., Bazri, K.E., Marref, S.E., Lekmine, S., & Amari, A. (2021). Effects of single and combined olive mill wastewater and olive mill pomace on the growth, reproduction, and survival of two earthworm species (Aporrectodea trapezoides, Eisenia fetida). *Applied Soil Ecology, 168,* 104123.

Mekki, A., Dhouib, A., & Sayadi, S. (2007). Polyphenols dynamics and phytotoxicity in a soil amended by olive mill wastewaters. *Journal of Environmental Management, 84,* 134–140.

Mekki, A., Dhouib, A., & Sayadi, S. (2009). Evolution of several soil properties following amendment with olive mill wastewater. *Progress in Natural Science, 19,* 1515–1521.

Melgar, J. C., Mohamed, Y., Serrano, N., García-Galavís, P. A., Navarro, C., Parra, M. A., ... & Fernández-Escobar, R. (2009). Long-term responses of olive trees to salinity. *Agricultural Water Management, 96*(7), 1105-1113.

Mohawesh, O., Al-Hamaiedeh, H., Albalasmeh, A., Qaraleh, S., & Haddadin, M. (2019). Effect of olive mill wastewater (OMW) application on soil properties and wheat growth performance under rain-fed conditions. *Water, Air, & Soil Pollution, 230,* 160.

Montemurro, F., Diacono, M., Vitti, C., & Ferri, D., (2011). Potential use of olive mill wastewater as amendment: crops yield and soil properties assessment. *Communications in soil science and plant analysis 42,* 2594–2603.

Moraetis, D., Stamati, F. E., Nikolaidis, N. P., & Kalogerakis, N. (2011). Olive mill wastewater irrigation of maize: Impacts on soil and groundwater. *Agricultural Water Management, 98*(7), 1125-1132.

Nadal-Romero, E., Cammeraat, E., Serrano-Muela, M.P., Lana-Renault, N., & Regüés, D. (2016). Hydrological response of an afforested catchment in a Mediterranean humid mountain area: a comparative study with a natural forest. *Hydrological Process, 30,* 2717–2733.

Olsen, S. (1982). Phosphorous. *Methods of soil analysis,* 403–430.

Paredes, M.J., Moreno, E., Ramos-Cormenzana, A., & Martinez, J. (1987). Characteristics of soil after pollution with waste waters from olive oil extraction plants. *Chemosphere, 16,* 1557–1564.

Pedrero, F., & Alarcón, J.J. (2009). Effects of treated wastewater irrigation on lemon trees. *Desalination, 246,* 631–639. https://doi.org/10.1016/j.desal.2008.07.017

Pedrero, F., Allende, A., Gil, M.I., Alarcón, J.J., 2012. Soil chemical properties, leaf mineral status and crop production in a lemon tree orchard irrigated with two types of wastewater. *Agricultural Water Management, 109,* 54–60.

Piotrowska, A., Rao, M.A., Scotti, R., & Gianfreda, L. (2011). Changes in soil chemical and biochemical properties following amendment with crude and dephenolized olive mill waste water (OMW). *Geoderma, 161,* 8–17.

Regni, L., Pezzolla, D., Ciancaleoni, S., Marozzi, G., Albertini, E., Gigliotti, G., & Proietti, P. (2021). Long-term effects of amendment with Olive Mill Wastewater on soil chemical properties, microbial community, and olive tree vegetative and productive activities. *Agronomy, 11,* 2562.

Rodier, J., Bazin, C., Broutin, J.P., Chambon, P., Champsaur, H., & Rodi, L. (2009). Water analysis, 9th edit. Dunod, Paris, France 1579.

Roig, A., Cayuela, M.L., & Sánchez-Monedero, M.A. (2006). An overview on olive mill wastes and their valorisation methods. *Waste Management, 26,* 960–969.

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.

Sainju, U. M., Liptzin, D., & Jabro, J. D. (2022). Relating soil physical properties to other soil properties and crop yields. *Scientific Reports*, 12(1), 22025.

Shabir, S., Ilyas, N., Saeed, M., Bibi, F., Sayyed, R.Z., & Almalki, W.H. (2023). Treatment technologies for olive mill wastewater with impacts on plants. *Environmental Research, 216,* 114399.

Shahalam, A., Zahra, B.M.A., & Jaradat, A. (2016). Wastewater Irrigation Effect on Soil, Crop and Environment: a Pilot Scale Study at Irbid, Jordan.

Sierra, J., Martí, E., Garau, M.A., & Cruañas, R. (2007). Effects of the agronomic use of olive oil mill wastewater: field experiment. *Science of the total Environment, 378,* 90–94.

Singh, K., Vasava, H. B., Snoeck, D., Das, B. S., Yinil, D., Field, D., & Panigrahi, N. (2019). Assessment of cocoa input needs using soil types and soil spectral analysis. *Soil Use and Management, 35*(3), 492-502. https://doi.org/10.1111/sum.12499.

Singleton, V.L., & Rossi, J.A., 1965. Colorimetry of total phenolics with phosphomolybdic-phosphotungstic acid reagents. *American Journal of Enology and Viticulture, 16,* 144–158.

Steinmetz, Z., Kurtz, M. P., Dag, A., Zipori, I., & Schaumann, G. E. (2015). The seasonal influence of olive mill wastewater applications on an orchard soil under semi-arid conditions. *Journal of Plant Nutrition and Soil Science*, 178(4), 641-648.

Tamimi, N., Diehl, D., Njoum, M., Marei, A., & Schaumann, G. E. (2016). Effects of olive mill wastewater disposal on soil: Interaction mechanisms during different seasons. *Journal of Hydrology and Hydromechanics, 64*(2), 176-195.

Thielmann, J., Kohnen, S., & Hauser, C. (2017). Antimicrobial activity of *Olea europaea* Linné extracts and their applicability as natural food preservative agents. *International Journal of Food Microbiology, 251*, 48-66.

Vaz, T., Quina, M.M., Martins, R.C., & Gomes, J. (2024). Olive mill wastewater treatment strategies to obtain quality water for irrigation: A review. *Science of The Total Environment,* 172676.

Vella, F.M., Galli, E., Calandrelli, R., Cautela, D., & Laratta, B. (2016). Effect of olive mill wastewater spreading on soil properties. *Bulletin of Environmental Contamination and Toxicology, 97,* 138–144.

Walkley, A., & Black, I.A. (1934). An examination of the Degtjareff method for determining soil organic matter, and a proposed modification of the chromic acid titration method. *Soil Science, 37,* 29–38.

Wang, J., Wang, G., & Wanyan, H. (2007). Treated wastewater irrigation effect on soil, crop and environment: Wastewater recycling in the loess area of China. *Journal of Environmental Science, 19,* 1093–1099. https://doi.org/10.1016/S1001-0742(07)60178-8

Waterman, P.G., Mole, S., 1994. Analysis of phenolic plant metabolites.

Yang, Y., Wu, J., Du, Y. L., Gao, C., Tang, D. W., & van der Ploeg, M. (2022). Effect on soil properties and crop yields to long-term application of superabsorbent polymer and manure. *Frontiers in Environmental Science*, 10, 859434.

Zema, D.A., Carrà, B.G., Sorgonà, A., Zumbo, A., Lucas-Borja, M.E., Miralles, I., Ortega, R., Soria, R., Zimbone, S.M., & Calabrò, P.S. (2023). Sustainable Use of Treated Municipal Wastewater after Chlorination: Short-Term Effects on Crops and Soils. *Sustainability, 15,* 11801. https://doi.org/10.3390/su151511801

Zema, D.A., Esteban Lucas-Borja, M., Andiloro, S., Tamburino, V., & Zimbone, S.M. (2019). Short-term effects of olive mill wastewater application on the hydrological and physico-chemical properties of a loamy soil. Agricultural Water Management, 221, 312–321. https://doi.org/10.1016/j.agwat.2019.04.011.

Zenjari, B., & Nejmeddine, A. (2001). Impact of spreading olive mill wastewater on soil characteristics: laboratory experiments. *Agronomie, 21,* 749–755.