Short-term effects of olive mill wastewater application on the hydrological and physico-chemical properties of a loamy soil

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Abstract

Wastewater from the olive oil industry is often spread on soils, but this practice requires caution because of the possible harmful hydrological and physico-chemical effects on soils treated. While research has mainly focussed on the long-term changes in soil properties, very few studies refer to short-term effects, which appear a few weeks or months after Olive Mill Wastewater (OMW) application. Mediterranean areas are particularly prone to runoff and soil erosion risks, and the evaluation of the short-term effects of OMW on hydraulic conductivity and other physico-chemical properties of soils in this area are scarce. To fill this gap, this study has evaluated the effects of OMW spreading on the hydrology of a loamy soil at one and three weeks after application. More specifically, irrigation tests with OMW were carried out in olive field plots in Calabria, Southern Italy; the infiltrability and physico-chemical properties of treated soils were compared with unirrigated soils and soils irrigated with clean water, such as groundwater.

A temporary reduction in the soil infiltration rate was detected immediately after OMW irrigation, although it was followed by a significant increase after three weeks. In the topsoil, irrigation with OMW produced an increase in phosphorous, potassium and organic matter contents as well as a higher stability of aggregates. In the deeper layer of soil treated with OMW the contents of organic matter and nutrients as well as of polyphenols were lower than in the unirrigated plot. Potassium, calcium and sodium contents were higher in the soil irrigated with OMW both in topsoil and deeper layers. Principal Component Analysis highlighted a noticeable influence of treatments on soil micronutrient contents, Cation Exchange Capacity and the stability of aggregates with slight differences between topsoil and deeper soil layers. Conversely, the concentration of organic matter, polyphenols and nutrients is noticeably stratified in both the topsoil and the deeper layers regardless the type of treatment.

Keywords: *infiltration rate; organic matter; soil physico-chemical changes; wastewater irrigation; soil aggregate stability; runoff and erosion risk.*

1. Introduction

The extraction process of olive oil requires the addition of a high volume of water, a large share of which becomes waste (Aktas et al., 2001; Mechri et al., 2007; Sellami et al., 2008). This oil mill effluent, known as Olive Mill Wastewater (OMW), contains the water added during the process, the water and pulp contents of olives, and a stable emulsion of oil residues (Paredes et al., 2002; Brunetti et al., 2007). Generally, OMW shows high concentrations of organic matter, suspended solids and inhibiting compounds, such as polyphenols; the content in mineral elements, such as potassium, phosphorus and calcium, is lower (Amaral et al., 2008; Di Serio et al., 2008). Because of these peculiar characteristics, uncontrolled disposal of OMW poses serious environmental risks to water, soil and air, such as the pollution of surface and ground water bodies, degradation of soil properties, and foul-smelling emissions (Dermeche et al., 2013; Chaari et al., 2015). The most dangerous OMW properties are the acidic pH and the high contents of both organic matter and phenols (Dermeche et al., 2013; Chaari et al., 2015; Aggoun et al., 2016).

To reduce the environmental drawbacks linked to OMW disposal, several physico-chemical - e.g. evaporation, reverse osmosis, filtration, oxidation, thermal drying - or biological - e.g. aerobic and anaerobic treatments, composting, phyto-depuration - methods have been proposed for OMW treatment (Dourou et al., 2016). However, until now, their economic and technological application is in most cases limited (Chartzoulakis et al., 2010).

In contrast to the usual OMW treatments, several studies have suggested the controlled application of OMW to cultivated soil. According to these studies, OMW can be used as a cheap soil conditioner and/or fertilizer (Saadi et al., 2007; Caputo et al., 2013; Ayoub et al., 2014; Barbera et al., 2014), and represents an additional water and nutrient resource for fertilisation/irrigation of Mediterranean olive groves, affected by a chronic water and organic matter scarcity (Di Bene et al., 2013; Ayoub et al., 2014; Chaari et al., 2015). In this practice, called "OMW land treatment and utilisation", OMW is applied to bare or cultivated soil (Cabrera et al., 1996; Sierra et al., 2001; Kapellakis et al., 2008; Mechri et al., 2008).

However, OMW land treatment must be practised with caution, since OMW application may induce serious environmental damage due to its toxicity towards plants and bacteria living in soils (Moreno et al., 1987; Mekki et al., 2006; Barbera et al., 2014). Moreover, the agronomic use of OMW is limited by other constraints, such as oil and grease (Amaral et al., 2008; Ouzounidou et al., 2008), suspended solids (Paredes et al., 1987; Roig et al., 2006; Chaari et al., 2015) as well as high salinity and acidity (Barbera et al., 2014; Cavallaro et al., 2014). Therefore, the impacts of OMW application on crops and on the chemical, physical and biological properties of soils must be further studied and tested in field, since little is known about these effects (Aggelis et al., 2003; Mekki et al., 2008; Mekki et al., 2009). A comprehensive review was carried out by Mekki et al. (2006) and more recently Barbera et al. (2013). In general, due to the variability of OMW application rates and properties of receiving soils, literature data on OMW effects on soil properties is not unanimous and, in many cases, even contradictory (Chartzoulakis et al., 2010). In addition, most of the studies reported in the scientific literature have mainly evaluated the long-term effects on soil properties - that is, on a yearly or decade scale - with contrasting results (Greco et al., 2006; López et al., 1996; Piotrowska et al., 2006; 2011).

Compared to studies on the long-term effects, those on the short-term effects are far fewer (e.g. Levi-Minzi et al., 1992; Sierra et al., 2007; Di Serio et al., 2008; Di Bene et al., 2013; Piotrowska et al., 2011). The evaluation of the effects of OMW on the chemical, physical and biological soil properties performed some weeks or months after OMW application on soil are equally important, since many adverse effects may be present only in this period. For instance, Sierra et al. (2007) detected increases in soil salinity and in content of phenolic compounds four months after OMW application. A decrease in soil pH and polyphenol (PP) content were claimed by Di Serio et al. (2008) in 2-month field experiments. Di Bene et al. (2013) and Caruso et al. (2018) suggested strict control of OMW use as an organic conditioner given the negative effects on soil properties such as the increase in PP and electrical conductivity and decrease in fungal communities five days after OMW application). These early adverse effects become more negligible as time passes. As a general response to OMW application, Piotrowska et al. (2011) observed sudden changes in some biological properties of the soil immediately after application and in the following 14 days. By contrast, no differences in COD (Chemical Oxygen Demand), phenol content and electrical conductivity of soils were detected three months after OMW application by Levi-Minzi et al. (1992) compared to untreated soils.

Furthermore, few studies have focused on the short term hydrological effects of OMW application. Thus, the time evolution of infiltrability on soils treated with OMW is also not completely understood and literature data about the effects of OMW on water infiltration and hydraulic conductivity of soil are contrasting (Barbera et al., 2013). Much caution should be paid to the possible changes in the hydrological properties of receiving soils, such as infiltration rates. In Mediterranean areas particularly prone to runoff and soil erosion risks (Zema et al., 2014; Fortugno et al., 2017; Lucas-Borja et al., 2018; Zema et al., 2018), OMW should not be applied to soils during the wet period; a decrease in soil infiltrability after OMW land treatment may worsen the hydrological response to intense storms on the steep olive groves of some Mediterranean areas (e.g. Southern Italy, Tamburino et al., 1999; Bombino et al., 2010; Spain, Gomez et al., 2009; Burguet et al., 2016). Therefore, more research may help to better understand the changes of infiltrability and physico-chemical properties of soils immediately or few weeks after receiving OMW.

To fill this gap, this study evaluates the short-term effects of OMW spreading on soil hydrology in the immediate weeks after application. More specifically, irrigation tests with OMW have been carried out in olive field plots at an experimental site in Calabria, Southern Italy, and the infiltrability and physico-chemical properties of treated soils were compared with unirrigated soils and soils irrigated with clean water. We hypothesized that the short-term effects of OMW application are not as beneficial as those reported after long-term monitoring (e.g. Mekki et al., 2006; 2007; Mechri et al., 2008; Moraetis et al., 2011). This experimental study aims to address the insufficient literature available on this topic, to either confirm or reject this hypothesis.

2. Materials and methods

2.1. Study area

The tests were carried in plots on an olive farm, 180 m above mean sea level, geographical coordinates 38.9685N, 16.2883E, in the territory of Lamezia Terme, Calabria, Southern Italy (Figure 1a). Olive grove soils were characterized from forty-five samples collected at two different depths, 0-5 and 30-40 cm below ground. Soil showed on average 34% of sand, 36% of silt and 31% of clay (w/w) and lack of skeleton, and was classified as Alfisol, loamy soil with moderately fine texture, according to the USDA Soil Taxonomy.

2.2. Experimental design

In the olive grove, three plots, each one including 18 seven-year-old olive trees - cultivar *Carolea* - planted at 5 x 4 m, were chosen (Figure 1b). Each plot covered an area of 360 m^2, 24-m long by 15-m wide. Within each plot, three sample areas were chosen (Figure 1b), of which:

- one area was not irrigated - henceforth indicated as "No irrigation" - and assumed as control;
- a second area was treated in March with OMW, henceforth indicated as "Irrigation with OMW";
- a third area was treated in March with clean water, henceforth indicated as "Irrigation with CW".

At one and three weeks after irrigation, the sample areas in the three plots were subject to infiltration tests. After eight weeks the main chemical-physical properties of soil in each sample area of the three plots were evaluated at two different profile depths, 0-5 and 30-40 cm (Figure 1b). All measurements were carried out in triplicate.

2.3. Irrigation equipment and methods

The micro-irrigation system consisted of a 40-mesh disk filter, a main PVC pipeline, 75 mm in diameter, and a secondary low-density polyethylene pipeline, 32 mm, supplying eight distributing lines, 16 mm, provided with drippers of long-path type. Each olive tree was irrigated by two drippers distributing 8 L h^{-1} at distance of 1 m. The irrigation water was pressured by an electric pump of nominal power of 4 kW with pressure manually setup through a by-pass set at a pressure of 1-1.1 bar. Further details over the irrigation system are reported in the paper by Capra et al. (2005).

The hydraulic load, equal for plots irrigated with OMW and CW, was 75 m^3 ha^{-1}, equivalent to 150 L per olive tree in 10 hours, and 33 mm on the area of effective infiltration. The hydraulic load of OMW supply is close to the maximum limit permitted by the Italian law n. 574/96, equal to 80 m^3 ha^{-1} per year.

2.4. Water analysis

OMW was supplied from a continuous three-phase oil extraction system and stored in the period November-March in a 180 m^3 open concrete tank. CW was pumped from a well.

Analysis of OMW and CW was carried out in triplicate on water samples following the Italian standards (APAT IRSA-CNR, 2003), which refer to the common international methods (APHA-AWWA-EF, 1998; ASTM, 1981; EPA, 1974). OMW was characterized at the beginning, during, and at the end of the watering, while CW was sampled at the beginning of watering (Table 1).

2.5. Infiltration tests

Measurements of soil infiltration rates were performed by a double-cylinder infiltrometer, consisting of two coaxial cylinders having inner and outer diameters of 320 and 570 mm, respectively, and height of 300 mm and driven into the soil to a depth of 150 mm. The test measured the time needed for the infiltration of 20 mm of water in the cylinders filled with 50-70 mm of CW. The infiltration test was repeated until three equal time measurements had been recorded between filling operations. The ratio between the water depth of 20 mm and the time recorded for water infiltration gave the soil infiltration rate. The infiltration measurements were made in three randomly chosen points of each plot. Soil samples with cracks or pebbles, which could lead to unrealistic measurements, were excluded.

2.6. Soil analysis

The following determinations on the soil fraction finer than 2 mm (after sample air-drying and sieving) were performed: pH (by portable electrochemical instrument Hach Lange HQ30d), organic matter content (OM) (Walkley-Black method, 1934), total nitrogen (TN) (Bremmer & Mulvaney, 1982), phosphorous (P) (Olsen method, 1954) and potassium/magnesium/sodium/calcium contents (K, Mg, Na, Ca exchangeable cations) (using the barium-chloridetriethanolamine method, Thomas, 1982); based on these data, the Cation Exchange Capacity (CEC) was calculated. Furthermore, the following properties were evaluated from the 1:5 soil/water extract, obtained by a 30-minute shaking: phenolic compounds (PP) (using the Folin-Ciocalteau reagent), volatile acids (VolAcid) (by titulation, Di Lallo and Alberton, 1961) and aggregate stability index (ASIndex) (by wet sieving, Pagliai, 1997).

2.7. Statistical analysis

First, the physico-chemical properties of the soil were analysed by a two-way ANOVA, using as factors the treatment (that is, no irrigation or irrigation with OMW or irrigation with CW), soil sampling depth (that is, 0-5 or 30-40 cm), and the interactions between treatment and soil sampling depth. The post hoc test applied was Fisher's least significant difference at $p < 0.05$ level of significance.

Then, Tukey's test at $p < 0.05$ was also used to evaluate the statistical significance of the differences in soil infiltration rates and physico-chemical changes between pairs of treaments; the latter comparisons were performed separately at the two sampling depths The same test was preliminarily applied to physico-chemical properties of irrigation waters used for the treatments (that is, OMW and CW).

Finally, Principal Component Analysis (PCA) was performed on a Spearman rank correlation matrix of the physico-chemical properties of soils sampled at the two depths after treatments to reduce the dimensionality of the data set.

In order to satisfy the assumptions of the statistical tests, that is the equality of variance and normal distribution, the data were subjected to normality test or were square root-transformed whenever necessary. The statistical analyses were carried out with the Statgraphics Centurion and XLSTAT software.

3. Results

3.1. Characterisation of OMW and CW

OMW was slightly acid (mean value of pH equal to 5.73) and had a moderate Electrical Conductivity (EC) (average 1584.33 μS cm^{-1}, indicating a moderate salt content) (Table 1). The mean Total Suspended Solids (TSS) and Total Settleable Solids (TSeS) concentrations (130.67 and 14.53 mg L^{-1}, respectively) were relatively high. COD was on average 59 g L^{-1} and TN was 134,97 mg L^{-1} (Table 1). The PP concentration (38.03 mg L^{-1}) was close to the lower value typical of the fresh OMW (0.5 - 24 g L^{-1}, Borja et al., 2006; Gonzalez-Lopez et al., 1994).

CW used in the other irrigated plots was poor in TSS and TSeS (18 and 0.03 mg L^{-1}) and with neutral pH (6.73). The contents of OM (on average 0.03 g L^{-1}) as well as micro- and macro-nutrients were much lower than OMW, except for P and Na, while PP were absent (Table 1).

3.2. Changes of hydrological properties of the soil

Figure 2 illustrates the infiltration curves measured by the tests carried out by the infiltrometer. The initial infiltration rate was higher for plots irrigated with OMW and lower for irrigation with CW. The steady-state was achieved after about 6-7 hours in the unirrigated plots or plots irrigated with OMW, and after 12 hours in those irrigated with CW (Figure 2).

One week after irrigation the steady-state infiltration rate (3.31 mm h^{-1} in the case of OMW application and 2.22 mm h^{-1} for CW) was much lower compared to the unirrigated plot (42.3 mm h^{-1}). The plots irrigated with OMW showed a higher infiltration rate (by about 50%) compared to those irrigated with CW (Table 2).

Three weeks after irrigation, a shift of the steady-state infiltration rate was observed in the plots irrigated with OMW (20.74 mm h^{-1}, three times higher than measurements in plots irrigated with CW, 6.79 mm h^{-1}), although remaining lower than the values achieved in the unirrigated control plot (42.3 mm h^{-1}) (Table 2).

3.3. Changes of physico-chemical properties of the soil

Two-way ANOVA showed that the irrigation and the sampling depth factors separately influenced all the physico-chemical parameters of the soil, excluding Mg concentration for the irrigation factor and pH, CEC and ASIndex for soil depth. The interactions among these variability factors were always statistically significant except Ca, Na, CEC, VolAcid and ASIndex (Table 3).

In the topsoil, compared to the unirrigated soil, the plots treated with OMW showed a slight increase of pH (from 6.47 to 6.85). Irrigation with OMW increased the soil's content of OM (from 23.4 to 27.6 g kg^{-1}) and of all macro- and micronutrients with the exception of TN (1.6 g kg^{-1} for both) and Mg (210 and 206 g kg^{-1}, respectively). Due to the increase of OM content and cations, also ASIndex and CEC were higher in plots treated with OMW (34.97 and 13.83 Meq 100-g^{-1}, respectively) compared with unirrigated plots (20.33 and 10.37 Meq 100-g^{-1}, respectively). Also, the PP content of treated soil was higher (7.50 g kg^{-1}) than in the control plots (6.17 g kg^{-1}) (Table 3). Compared to the control plots, irrigation with CW decreased pH and the soil content of OM and micro/macro-nutrients, except sodium. The ASindex was higher than in unirrigated plots (21.6 against 20.33, respectively (Table 4).

In the deeper soil layer (30-40 cm), compared to control plots, irrigation with OMW increased pH (from 6.33 to 6.59) and decreased the soil content of OM, TN, P, Mg and PP; conversely, the content of K, Ca and Na as well as CEC and ASIndex were higher (Table 4). The same trend was detected in the plots irrigated with CW, but with a more noticeable decrease of K and PP contents (Table 3).

3.4. Statistical analysis

3.4.1. Correlation analysis

Positive and significant correlations were detected between OM and TN, P, K, Mg, PP and VolAcid. The pH of soil was also positively correlated with P, K, VolAcid and ASIndex. Moreover, TN and P were correlated positively with K, PP and VolAcid, and negatively with Na. Also, AsIndex showed a positive correlation with Na and Ca. Both VolAcid and AsIndex were not correlated with CEC and Mg. Finally, the correlation between CEC and K, Ca, Na was positive and significant (Table 5).

3.4.2. Principal Component Analysis (PCA)

By PCA two principal components, PC1 and PC2, were extracted, explaining about 75% of the total variance of the original variables, 47% for PC1 and 28% for PC2. Plotting the PCA scores on the two PCs allowed clustering six well-differentiated groups (Figure 3). The clustered groups were the following: i) irrigation with OMW + 30-40 sampling depth; ii) irrigation with OMW + 0-5 sampling depth; iii) irrigation with CW + 30-40 sampling depth; iv) irrigation with CW + 0-5 sampling depth; v) no irrigation + 30-40 sampling depth; and vi) no irrigation and 0-5 sampling depth (Figure 3). More specifically, as explained by the loadings of the soil parameters on the PCs, the organic matter (OM), the acidity (VolAcid) as well as the concentrations of nutrients (TN, P and K) and PP significantly influenced the first PC, while the concentrations of some micro-nutrients (Ca and Na) as well as the aggregate stability (ASIndex) mainly weighted on PC2 (Table 6 and Figure 3). Some of these correlations are in tune with those found by the study of Chaari et al. (2015), mainly those relating K and pH, OM and P.

From the multivariate statistical analysis by PCA two interesting considerations can be drawn. First of all, a gradient along the first principal component, mainly related to OM, TN, P, K and PP content, is evident among samples taken at different depths (Figure 3); in other words, these physico-chemical properties of topsoil are associated with positive values of PC1, except two samples irrigated with CW, while those of the deeper soil show negative values of PC1. Moreover, another evident gradient can be found along the second principal component, on which the concentration of Ca and Na as well as CEC and ASIndex mainly weigh (Figure 3); the different soil treatments are arranged along this gradient without noticeable differences between topsoil and deeper layers. The soil irrigated with OMW is associated with positive values of PC2, whereas the unirrigated soil has negative scores on the same PC.

4. Discussions

4.1. Characterisation of OMW and CW

The preliminary characterisation of residual (OMW) and clean (CW) waters used for olive grove irrigation showed a noticeable content~~s~~ of polluting compounds. Acid and saline water containing high concentrations of OM, macro- and micro-nutrients, and inhibiting compounds (PP) may alter in both short and long-term the physico-chemical and biological equilibrium of the treated soils (Moreno et al., 1987; Paredes et al., 1999; Piotrowska et al., 2011). For instance, when unstable organic matter is applied to soil, several negative effects can be detected on soil properties and plant growth, such as increased mineralization of soil organic carbon, anaerobic conditions, phytotoxicity, and immobilization of plant nutrients (Senesi, 1989; Cereti et al., 2004; Komilis et al., 2005; Brunetti et al., 2007).

4.2. Changes in hydrological properties of the soil

As regards the hydrological affects of OMW application, it is known that the hydraulic conductivity of saturated soils is generally reduced by irrigation with wastewater (Gharaibeh et al., 2007). This investigation showed that one week after watering a reduction of soil infiltration rate in the soil treated with OMW is evident. However, also the soil with CW irrigation showed a drastic decrease of infiltration. These reductions of infiltration rates detected in both irrigated soils may be due to soil particle movement, with surface pores clogging after irrigation; also the addition of exchangeable cations and high amounts of total suspended solids, of which our OMW is rich, and the accumulation of fatty substances of residual olive oil in OMW (Bhardwaj et al., 2008; Travis et al., 2008; Barbera et al., 2013) may have aggravated this behaviour of the top soil horizons.

After three weeks, steady-state infiltration rates of irrigated soils are lower than the values achieved in the unirrigated control plot. However, soils treated with OMW showed noticeable increases compared to soils irrigated with CW, whose infiltrability remains very low. Compared to the unirrigated plots, irrigation with CW showed a steady-state infiltration rate reduced by over 80%, while the value measured in the soil treated with OMW was 50% compared to the unirrigated plots. The higher infiltration rate of plots irrigated with OMW compared to those irrigated with CW probably depends on the soil particle aggregation effect provided by the addition of organic matter (López-Piñeiro et al., 2007). The results of this study are in agreement with the short-term reduction of soil infiltration rate reported by Andiloro et al. (2005) (Table 7). Moreover, Barbera et al. (2013) report that soil infiltration decreases approximately 20 days after OMW application and then gradually increases with a trend likely related to the biological decomposition of the fatty substances. The short-term reduction of soil infiltration rate detected in our study confirms the working hypothesis that the OMW application may worsen the hydrological response of soil immediately after its application. Therefore, the reduction in soil infiltrability suggests caution in irrigation of agricultural land, since it may increase runoff and erosion processes. However, the noticeable improvement of the hydrological response of soils treated with OMW compared to the decreased infiltration rates shown by soils irrigated by CW confirms the benefits of OMW application against the possible hydrological risks occurring in steep slopes after intense storms.

4.3. Changes in physico-chemical properties of the soil

The action of organic matter seems to be responsible for the increase of the infiltration rate in soils treated with OMW compared to irrigation with CW. Organic matter improves the porosity (Cox et al., 1996) and other physical properties, such as the stability of aggregates (Table 3) within the topsoil (Chaney and Swift, 1984; Lado et al., 2004). Furthermore, the susceptivity of the soil to sealing is decreased (Le Bissonais, 1996). The results confirmed the high correlation between organic matter and the soil infiltration rate, also detected by Mbagwu and Auerswald (1999, 0.55, significant at p level < 0.05).

Contrasting effects in the analysed soil properties were found in the treated plots and the investigated soil depths. Many of these contrasts were significant and mutually dependent, as two-way ANOVA demonstrated.

At both investigated soil depths, OMW application induced a slight increase of pH values, in spite of the OMW slight acidity. Presumably, the high buffer capacity of the soil did not decrease soil pH after the OMW application. This beneficial action seems to assure a soil tolerance in receiving acid wastewater. Therefore, in the short term no detrimental effects to pH were detected in the experimental soil.

Literature reports contrasting results about changes in soil pH after OMW application. Most studies report pH decreases due to irrigation with OMW. This was detected both in the short term~~s~~ (within 5-6 months, Zenjari and Nejmeddine, 2001; Sierra et al., 2007; Di Serio et al., 2008; Brunetti et al., 2007) and many months after irrigation (Chaari et al., 2015) (Table 7). According to these authors, soil acidity slightly decreases in the upper layer immediately after OMW application, since OMW had a low pH (Di Serio et al., 2008). Conversely, others authors reported, as in our study, slight increase of pH, when soils were irrigated with OMW (Di Bene et al., 2013, at six months in the topsoil; Piotrowska et al., 2011, after seven weeks; Chartzoulakis et al., 2005, in the long term) (Table 7).

An increase in organic matter content was detected in the topsoil, which supports the working hypothesis that OMW land application has a beneficial effect on soil properties also in the short term~~s~~. Except Sierra et al. (2007) and Di Bene et al. (2013, on a sandy loam soil), all the authors reported increases in organic matter or carbon content of the topsoil, after OMW application, both in short (Zenjari and Nejmeddine, 2001; Di Serio et al., 2008; Piotrowska et al., 2011; Brunetti et al., 2007) and in long terms (Andiloro et al., 2005; Mekki et al., 2006; Chartzoulakis et al., 2005; Chaari et al., 2015), because of the high organic matter content of the effluent (Table 7).

In the topsoil, a significant increase of potassium, and polyphenol contents, were also measured, while the total nitrogen did not change. For this nutrient, a slight reduction was instead recorded after irrigation with CW. Practically, all the previous literature report increases in the contents of nitrogen, phosphorous and potassium, with few exceptions. Among these latter, Sierra et al. (2007) and Piotrowska et al. (2011) detected reductions or no changes in nitrogen content of the topsoil in the short term~~s~~, while decreases in phosphorous (Sierra et al., 2007, after 120 days from irrigation, and Andiloro et al., 2005, after seven months) and potassium (Andiloro et al., 2005) concentrations were measured. The increases of potassium and phosphorous may well be due to the high phosphorous content of OMW and to the action of microflora, which affects the available potassium fraction in organic form within the effluent (Papini et al., 2000). The increase in the content of potassium and phosphorus enhances soil fertility and this suggests the viability of OMW use as soil fertiliser supposed in this study: therefore, the OMW application reduces the need for chemical fertilizers in spring with evident economic and ecological benefits (Di Serio et al., 2008).

The effects of the polyphenols are notable throughout three months after OMW application (Saviozzi et al., 1991; Saadi et al., 2007) and subsequently the content of phenols gradually decreases, since these compounds are broken down by specific bacteria, such as yeasts and fungi (Di Serio et al., 2008; Barbera et al., 2013). This study confirms the sharp increase in polyphenol content of the topsoil, which is in accordance with those literature studies carried out no later than 3-4 months after OMW applications (Zenjari and Nejmeddine, 2001, and Di Serio et al., 2008). After this period, Andiloro et al. (2005), Sierra et al. (2007) and Di Bene et al. (2013) showed that the polyphenol content is reduced both in the topsoil and in the deeper layer (Table 7). Sierra et al. (2007) recommended a limit of 180 m^3 ha^{-1} for OMW yearly application, because at doses over 360 m^3 ha^{-1} the phenolic content of soil may increase due to the temporal immobilization of nitrate, which may decrease plant production. Since the OMW application load of the present study (80 m^3 ha^{-1} $year^{-1}$) was much lower than the suggested limit, the phytotoxic and antibacterial actions of polyphenols, which represent the main limiting factor for spreading OMW, may be excluded in the short terms, considering the limited increase detected in the topsoil.

As regards the deeper soil layer (30-40 cm), the contents of organic matter, total nitrogen, phosphorous and polyphenols, basically similar in the plots irrigated both with OMW and CW, are lower than in the unirrigated control plot, presumably due to the leaching effects induced by water percolation into the soil's deeper layers. Conversely, a slight increase in the potassium content was measured after OMW application. According to Chartzoulakis et al. (2010), this allows a reduction of the potassium fertilizer added to soil treated with OMW.

At both the investigated profile depths of soil irrigated with OMW, a higher value of sodium content was found compared to the control. Here, careful attention should be paid, since excessive sodium application through wastewater may lead to soil structure decay (due to excessive alkalinity) and adverse effects on plant growth and yield (due to the increased osmotic potential).

Finally, Mahmoud et al. (2012) report that the effect of OMW application on aggregate stability has not been thoroughly studied, although this property is important in evaluating the hydrological behaviour and long-term crop productivity of soil (Letey, 1985). The aggregate stability index after irrigation with OMW is higher at both the investigated soil depths, compared to the control plot, and this may represent a beneficial effect of soil treatment with OMW. This increase may well be due to the higher soil aggregation induced by the OM content of wastewater (Table 7). This result contrasts with findings of Andiloro et al. (2005), who detected a reduction in aggregate stability in both the surface and deeper layers of the treated soil seven months after OMW application.

5. Conclusions

In this study, OMW was applied at a rate close to the maximum limit permitted by the Italian law, 80 m^3 ha^{-1} $year^{-1}$, on a loamy soil. The hydrological and physico-chemical effects were measured some weeks after OMW application and compared to untreated soils (control tests) and soils irrigated with CW. These short-term results of OMW application were not always as beneficial as those reported after long-term literature experiences.

As regards the hydrological aspects, OMW application temporarily reduced the soil infiltration rate immediately after irrigation. Subsequently, a significant increase, mainly at two or three weeks from watering, was noticed on the treated soils, although infiltration was lower than in the control. Therefore, steep soils should not be irrigated with OMW during or immediately before the wet season to avoid the risks of surface runoff and soil erosion.

A few weeks after irrigation, some of the monitored physico-chemical properties of the soil treated with OMW had improved (e.g. organic matter, phosphorous and potassium contents, soil aggregate stability) and the remaining properties were not negatively affected (e.g. pH, nitrogen and polyphenol content) by OMW application, compared to the unirrigated plots or those treated with CW. However, an excessive sodium concentration in applied OMW may lead to soil structure decay and plant development reduction.

PCA highlighted a noticeable influence of soil treatments on micro-nutrient contents, CEC and the stability of aggregates with slight differences between topsoil and deeper layers. Conversely, the concentration of organic matter, polyphenols and nutrients is noticeably stratified into the topsoil and the deeper layers regardless the type of treatment (irrigation with OMW or CW either no irrigation).

Overall, the experimental tests confirm that the effluents of olive oil mills, when used in addition to clear water for crop irrigation, may represent both a soil improvement and an important resource in Mediterranean agriculture. In such environmental contexts the availability of irrigation water is limited due to the semi-arid climate and competition with other uses, and soils are in general poor in fertilising compounds. Irrigation of soils with OMW reduces clean water demand and improves soil fertility, due to the addition of organic matter and nutrients. However, OMW should not be applied to soils during or immediately before the wet season. Finally, OMW land application avoids or, at least, limits the need for effluent depuration before its disposal, thus reducing the pollution risk to water bodies.

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TABLES

Table 1 - Main chemical-physical properties of OMW and CW used in the irrigation tests (on triple water samples).

Water parameter	Irrigation with OMW		Irrigation with CW	
	Mean	Std. Dev.	Mean	Std. Dev.
pH	5.73 a	0.06	6.47 a	0.06
TSS (mg L^{-1})	130.7 a	7.02	18.00 b	1.73
TSeS (mg L^{-1})	14.53 a	0.93	0.03 b	0.06
COD (g L^{-1})	59.00 a	3.25	0.03 b	0.06
TN (mg L^{-1})	135.0 a	4.96	8.80 b	0.44
P (mg L^{-1})	60.57 a	0.57	48.50 a	3.01
K (mg L^{-1})	694.0 a	5.36	0.57 b	0.12
Na (mg L^{-1})	30.40 a	1.75	20.17 a	0.70
Ca (mg L^{-1})	64.67 a	3.11	25.57 b	0.45
Mg (mg L^{-1})	36.90 a	1.15	16.30 b	1.15
PP (mg L^{-1})	38.03 a	0.21	0.00 b	0.00

Notes: TSS = Total Suspended Solids; TSeS = Total Settleable Solids; OM = Organic Matter; TN = Total Nitrogen; P = Phosphorous; K = Potassium; Na = Sodium; Ca = Calcium; Mg = Magnesium; PP = Polyphenols. Different letters indicate significant differences at $p < 0.05$ of Tukey's test.

Table 2 – Steady-state infiltration rates (in mm h^{-1}) measured in plots subject to treatments (mean ± standard deviation).

Treatment	**Time from irrigation**	
	One week after	Three week after
Irrigation with OMW	3.31 ± 0.34 a	20.74 ± 3.78 A
Irrigation with CW	2.22 ± 0.28 a	6.79 ± 0.65 B
No irrigation (control)	-	42.30 ± 6.15 C

Notes: Different letters indicate significant differences at $p < 0.05$ of Tukey's test; the values measured after three weeks are the final points of the infiltration curves of Figure 2.

Table 3 - Two-way ANOVA applied to physico-chemical parameters of soils subject to treatments.

Soil parameter	Treatment (no irrigation/irrigation with OMW/irrigation with CW)		Sampling depth (0-5/30-40 cm)		Interaction (treatment x sampling depth)	
	F-Ratio	*P-Value*	*F-Ratio*	*P-Value*	*F-Ratio*	*P-Value*
pH	53.11	<0.01	2.82	n.s.	10.14	<0.01
OM	155.85	<0.01	9.64	<0.01	33.41	<0.01
TN	494.72	<0.01	183.27	<0.01	78.45	<0.01
P	880.88	<0.01	661.27	<0.01	889.06	<0.01
K	1152.12	<0.01	661.71	<0.01	233.49	<0.01
Ca	30.33	<0.01	6.79	<0.01	0.21	n.s.
Na	644.96	<0.01	34.64	<0.01	1.95	n.s.
Mg	3.75	n.s.	35.25	<0.01	5.27	0.02
CEC	5.26	0.02	4.07	n.s.	0.59	n.s.
PP	15.37	<0.01	80.91	<0.01	11.99	<0.01
VolAcid	7.91	<0.01	5.06	0.04	2.84	n.s.
ASIndex	30.14	<0.01	0.82	n.s.	1.76	n.s.

Notes: OM = Organic Matter; TN = Total nitrogen; P = Phosphorous; K = Potassium; Ca = Calcium; Na = Sodium; Mg = Magnesium; CEC = Cation Exchange Capacity; PP = Polyphenols; VolAcid = Volatile acidity 1:5; ASIndex = Aggregate Stability Index; n.s. = not significant.

Table 4 - Main chemical-physical properties of soils subject to treatments (measurements taken eight weeks after irrigation).

	Sampling depth (cm)											
	0-5						30-40					
	Treatment											
Soil parameter	No irrigation		Irrigation with OMW		Irrigation with CW		No irrigation		Irrigation with OMW		Irrigation with CW	
	Mean	Std. Dev.	Mean	Std. Dev.	Mean	Std. Dev.	Mean	Std. Dev.	Mean	Std. Dev.	Mean	Std. Dev.
pH	6.47 a	0.03	6.85 a	0.15	6.33 a	0.02	6.33 a	0.02	6.59 b	0.09	6.29 a	0.02
OM ($g\ kg^{-1}$)	23.4 a	0.52	27.6 a	1.71	22.2 a	0.13	22.2 a	0.09	17.1 b	1.63	17.6 b	0.42
TN ($g\ kg^{-1}$)	1.60 a	0.05	1.60 a	0.03	1.46 a	0.02	1.46 a	0.02	1.00 b	0.02	1.03 b	0.03
P ($mg\ kg^{-1}$)	99.30 a	2.86	159.2 b	0.96	91.0 a	1.05	91.0 a	1.1	74.6 b	0.1	78.2 b	3.38
K ($mg\ kg^{-1}$)	434.2 a	11.02	814.2 b	11.25	405.6 a	16.81	405.6 a	16.8	442.9 a	15.7	192.8 b	21.26
Ca ($mg\ kg^{-1}$)	1168 a	128.1	1457 b	60.30	1111 a	8.87	1111 a	8.9	1361 b	78.1	1368 b	62.08
Na ($mg\ kg^{-1}$)	27.60 a	1.05	149.8 b	6.68	39.5 c	4.89	39.5 a	4.9	178.1 b	10.0	171.4 b	5.15
Mg ($mg\ kg^{-1}$)	210.7 a	6.92	205.5 a	19.53	200.2 a	2.77	200.2 a	2.8	161.9 b	10.7	164.3 b	17.78
CEC ($Meq\ 100\text{-}g^{-1}$)	10.37 a	1.07	13.83 b	0.75	10.0 a	1.79	10.0 a	1.8	11.9 a	0.7	11.2 c	1.21
PP ($mg\ kg^{-1}$)	6.17 a	1.00	7.50 b	0.52	5.23 a	0.68	5.23 a	0.68	3.23 b	0.52	2.77 c	0.23
VolAcid ($mg\ kg^{-1}$)	60.00 a	5.57	67.00 b	2.00	54.0 c	3.61	54.00 a	3.61	46.00 b	2.01	41.00 c	7.00
ASIndex(*)	20.33 a	1.22	34.97 b	3.26	21.6 a	1.56	21.6 a	1.6	33.0 b	4.0	28.0 c	2.10

Notes: (*) evaluated on the soil fraction finer than 0.2 mm as the percent ratio between the weight of soil particles remained aggregated after water mixing and the total height of soil particles; OM = Organic Matter; TN = Total nitrogen; P = Phosphorous; K = Potassium; Ca = Calcium; Na = Sodium; Mg = Magnesium; CEC = Cation Exchange Capacity; PP = Polyphenols; VolAcid = Volatile acidity 1:5; ASIndex = Aggregate Stability Index. Different letters indicate significant differences at $p < 0.05$ of Tukey's test.

Table 5 - Correlation matrix (significant correlations in bold at $p < 0.05$) among physico-chemical parameters of soils subject to treatments.

	pH	**OM**	**TN**	**P**	**K**	**Ca**	**Na**	**Mg**	**CEC**	**PP**	**VolAcid**	**ASIndex**
pH												
OM	0.31											
TN	0.29	**0.88**										
P	**0.77**	**0.75**	**0.67**									
K	**0.66**	**0.79**	**0.61**	**0.82**								
Ca	0.07	0.12	-0.26	0.14	0.34							
Na	0.11	-0.33	**-0.67**	-0.09	0.05	0.80						
Mg	-0.24	**0.75**	**0.71**	0.24	0.42	0.01	-0.44					
CEC	0.24	0.29	0.06	0.31	**0.49**	**0.62**	**0.53**	0.29				
PP	0.41	**0.92**	**0.90**	**0.77**	**0.79**	-0.04	-0.43	**0.66**	0.24			
VolAcid	**0.63**	**0.56**	**0.71**	**0.74**	**0.59**	-0.17	-0.39	0.31	0.23	**0.65**		
ASIndex	**0.60**	0.01	-0.31	0.41	0.43	**0.55**	**0.69**	-0.43	0.32	0.01	0.03	

Notes: OM = Organic Matter; TN = Total nitrogen; P = Phosphorous; K = Potassium; Ca = Calcium; Na = Sodium; Mg = Magnesium; CEC = Cation Exchange Capacity; PP = Polyphenols; VolAcid = Volatile acidity 1:5; ASIndex = Aggregate Stability Index.

Table 6 - Loading factors of parameters on the first Principal Components (PC1 and PC2) of PCA applied to physico-chemical parameters of soils subject to treatments.

Soil parameter	**Principal Component**	
	PC1	PC2
pH	0.239	0.261
OM	**0.388**	-0.043
TN	**0.376**	-0.229
P	**0.370**	0.151
K	**0.367**	0.204
Ca	0.016	**0.428**
Na	-0.142	**0.492**
Mg	0.256	**-0.216**
CEC	0.146	**0.324**
PP	**0.397**	-0.072
VolAcid	**0.334**	-0.033
ASIndex	0.043	**0.479**

Notes: values in bold are significant at $p < 0.05$; OM = Organic Matter; TN = Total nitrogen; P = Phosphorous; K = Potassium; Ca = Calcium; Na = Sodium; Mg = Magnesium; CEC = Cation Exchange Capacity; PP = Polyphenols; VolAcid = Volatile acidity 1:5; ASIndex = Aggregate Stability Index.

Table 7 - Comparison of the main physical and chemical properties of soils irrigated with OMW reported in literature (in brackets the measuring units of each parameter).

					Change in parameter (control → OMW treatment)								
Authors (country)	**OMW dose (per year)**	**Monitoring period**	**Soil texture**	**Layer depth (cm)**	**IR (mm h^{-1})**	**pH**	**OMC**	**TN**	**P**	**K**	**PP**	**ASindex**	**Note**
This study (Italy)	80 (m^3 ha^{-1})	2 months	Loam	0-5	42.3 → 20.7	6.47 → 6.85	23.4 → 27.6 (g kg^{-1})	1.60 → 1.60 (g kg^{-1})	99.3 → 159.2 (mg kg^{-1})	434 → 514 (mg kg^{-1})	6.17 → 7.50 (g kg^{-1})	20.33 → 34.97 (-)	-
				30-40	-	6.33 → 6.59	22.2 → 17.1 (g kg^{-1})	1.46 → 1.00 (g kg^{-1})	91.0 → 74.6 (mg kg^{-1})	406 → 443 (mg kg^{-1})	5.23 → 3.23 (g kg^{-1})	21.6 → 33.0 (-)	
Zenjari and Nejmeddine, 2001 (Morocco)	750 ml over a circle of 7-cm diameter	1 month	Clay	0-20	n.m.	8.1→ 6.1	13 → 44 (OC, g kg^{-1})	0.7 → 3.5 (g kg^{-1})	1.1 → 2.3 (g kg^{-1})	n.m.	20 → 410 (mg kg^{-1})	n.m.	-
				20-40		8.1 → 7.8	10 → 32 (OC, g kg^{-1})	0.2 → 4.5 (g kg^{-1})	0.8 → 2.0 (g kg^{-1})		20 → 290 (mg kg^{-1})		
Piotrowska et al., 2011 (Morocco)	80 (m^3 ha^{-1})	42 days	Sandy clay loam	0-10	n.m.	8.3 → 8.7	13.9 → 15.58 (TOC, g kg^{-1})	1.6 → 1.6 (g kg^{-1})	34.3 → 35.5 (mg kg^{-1})	300 → 916 (mg kg^{-1})	n.m.	n.m.	-
Di Serio et al., 2008 (Italy)	160 (m^3 ha^{-1})	2 months	Sandy loam	10-20	n.m.	6.9 → 5.9	14.0 → 19.3 (g kg^{-1})	n.m.	13.90 → 81.15 (mg kg^{-1})	129.40 → 289.20 (mg kg^{-1})	1.4 → 2.4 (mg kg^{-1})	n.m.	-
				20-40		6.4 → 6.6	13.6 → 18.1 (g kg^{-1})		44.60 → 47.70 (mg kg^{-1})	74.70 → 129.20 (mg kg^{-1})	1.3 → 2.8 (mg kg^{-1})		
Sierra et al., 2007 (Spain)	360 (m3/ha)	120 days	Clay loam	0-20	n.m.	8.6 → 8.0	17.3 →15.3 (OC, g kg^{-1})	2.15 → 2.00 (g kg^{-1})	480 → 450 (g kg^{-1})	n.m.	110 → 25 (mg/kg)	n.m.	-
Brunetti et al., 2007 (Italy)	300 (m^3 ha^{-1})	4 months	Sandy loam	0-20	n.m.	8.0 → 7.8	10.3 → 12.4 (TOC, g kg^{-1})	1.0 → 1.1 (g kg^{-1})	33 → 52 (mg kg^{-1})	186 → 326 (mg kg^{-1})	n.m.	n.m.	Lagooned OMW
	600 (m^3 ha^{-1})					8.0 → 7.9	10.3 → 14.5 (TOC, g kg^{-1})	1.0 → 1.1 (g kg^{-1})	33 → 68 (mg kg^{-1})	186 → 460 (mg kg^{-1})			
Di Bene et al., 2013 (Italy)	80 (m^3 ha^{-1})	6 months	Silty clay loam	0-20	n.m.	7.78 → 8.15	11.21 → 11.41 (OC, g kg^{-1})	1.37 → 1.44 (g kg^{-1})	1.43 → 1.83 (g kg^{-1})	227.3 → 304.5 (mg kg^{-1})	4.00 → 2.25 (mg kg^{-1})	n.m.	-
			Sandy loam			7.77 → 7.75	10.04 → 8.31 (OC, g kg^{-1})	1.17 → 1.00 (g kg^{-1})	1.73 → 0.87 g kg^{-1})	285.3 → 373.7	6.33 → 6.00 (mg kg^{-1})		

										(mg kg^{-1})			
Andiloro et al., 2005 (Italy)	80-300 (m^3 ha^{-1})	7 months	Loam	0-5	8.73 → 4.15	n.m.	30 → 9 (g kg^{-1})	n.m.	140 → 80 (mg kg^{-1})	1000 → 700 (mg kg^{-1})	30 → 10 (mg g^{-1})	18 → 9 (-)	-
				30-40	-	n.m.	20 → 20 (g kg^{-1})		80 → 60 (mg kg^{-1})	1000 → 700 (mg kg^{-1})	30 → 20 (mg g^{-1})	14 → 11 (-)	
Mekki et al., 2006 (Tunisia)	200 (m^3 ha^{-1})	1 year	Sand	0-10	n.m.	7.9 → 7.4	2.0 → 17.0 (TC, g kg^{-1})	0.23 → 0.91 (g kg^{-1})	0.02 → 0.08 (g kg^{-1})	0.14 → 0.80 (g kg^{-1})	n.m.	n.m.	-
Chartzoulakis et al., 2005 (Greece)	252-420 (m^3 ha^{-1})	3 years	Loam	0-25	n.m.	6.8 → 6.9	9.2 → 10.1 (g kg^{-1})	0.79 → 1.30 (%)	70.8 → 79.0 (mg kg^{-1})	0.0 → 17.5 (mg kg^{-1})	n.m.	n.m.	-
				25-50		6.8 → 6.9	8.0 → 8.8 (g kg^{-1})	6.8 → 8.9 (g kg^{-1})	40.2 → 50.5 (mg kg^{-1})	0.0 → 7.1 (mg kg^{-1})			
Chaari et al., 2015 (Tunisia)	200 (m^3 ha^{-1})	9 years	Sand	0-20	n.m.	7.88 → 7.83	0.68 → 5.0 (g kg^{-1})	150 → 343 (mg kg^{-1})	52.5 → 77 (mg kg^{-1})	90 → 900 (mg kg^{-1})	2835 → 4581 (mg kg^{-1})	n.m.	-
				40-80		8.16 → 8.17	0.70 → 1.20 (mg kg^{-1})	195 → 190 (mg kg^{-1})	50 → 48 (mg kg^{-1})	80 → 85 (mg kg^{-1})	3010 → 3490 (mg kg^{-1})		

Notes: IR = infiltration rate; n.m. = not measured; OC = organic carbon; TC = total carbon; TOC = total organic carbon; OM = Organic Matter; TN = Total nitrogen; P = Phosphorous; K = Potassium; PP = Polyphenols; ASIndex = Aggregate Stability Index.

FIGURES

Figure 1 - Geographic location of the investigated farm (a) and scheme of the experimental tests (b).

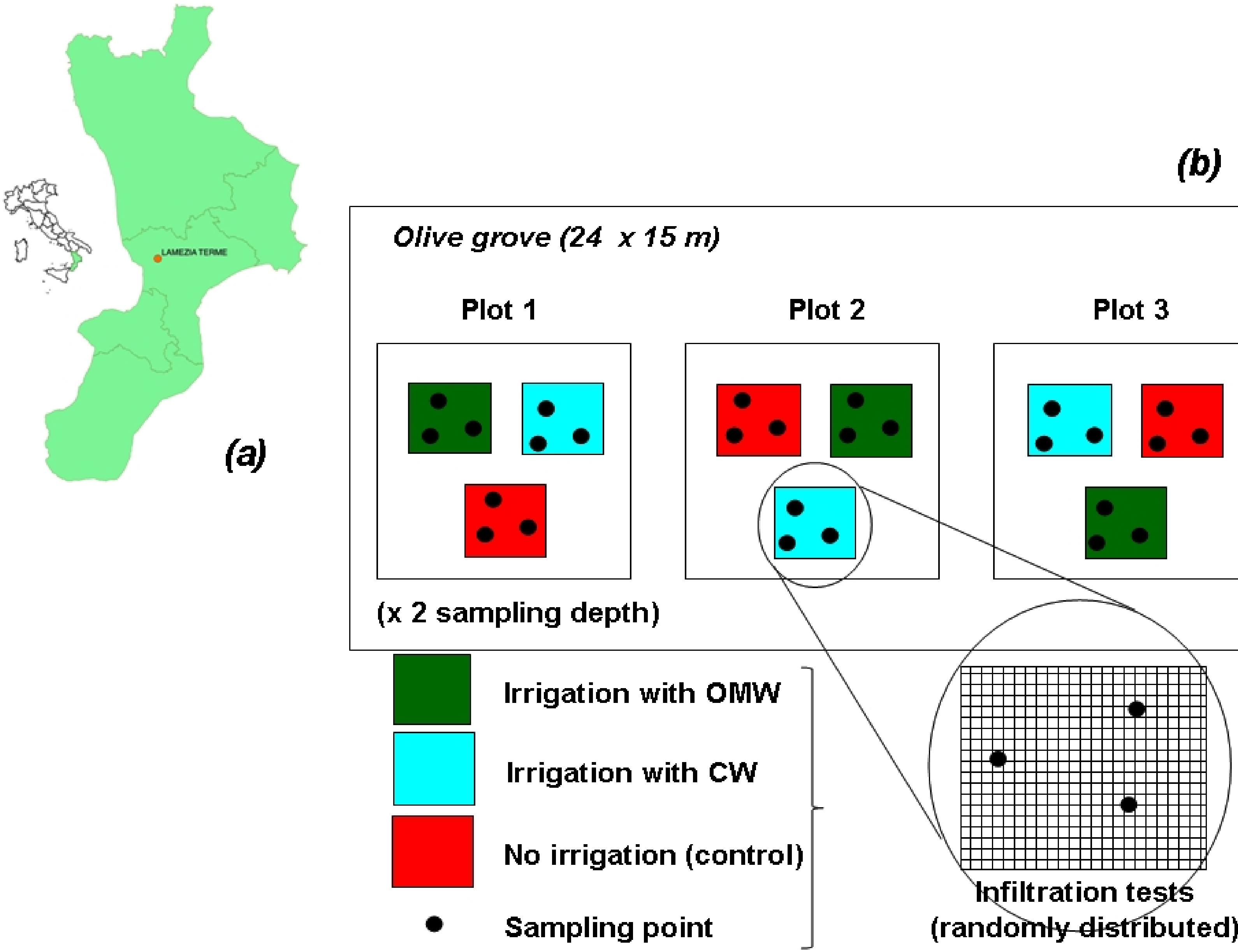
In three plots (1, 2 and 3) three sample areas (in green, cyan and red) were identified, of which one area was not irrigated (control) and the two other areas were subject to treatment with OMW or clean water, respectively. In each plot, at one and three weeks after irrigation, infiltration tests were carried out (black points) and, after eight weeks, the main chemical-physical properties of soil at depths of 0-5 and 30-40 cm. All measurements were carried out in triplicate.

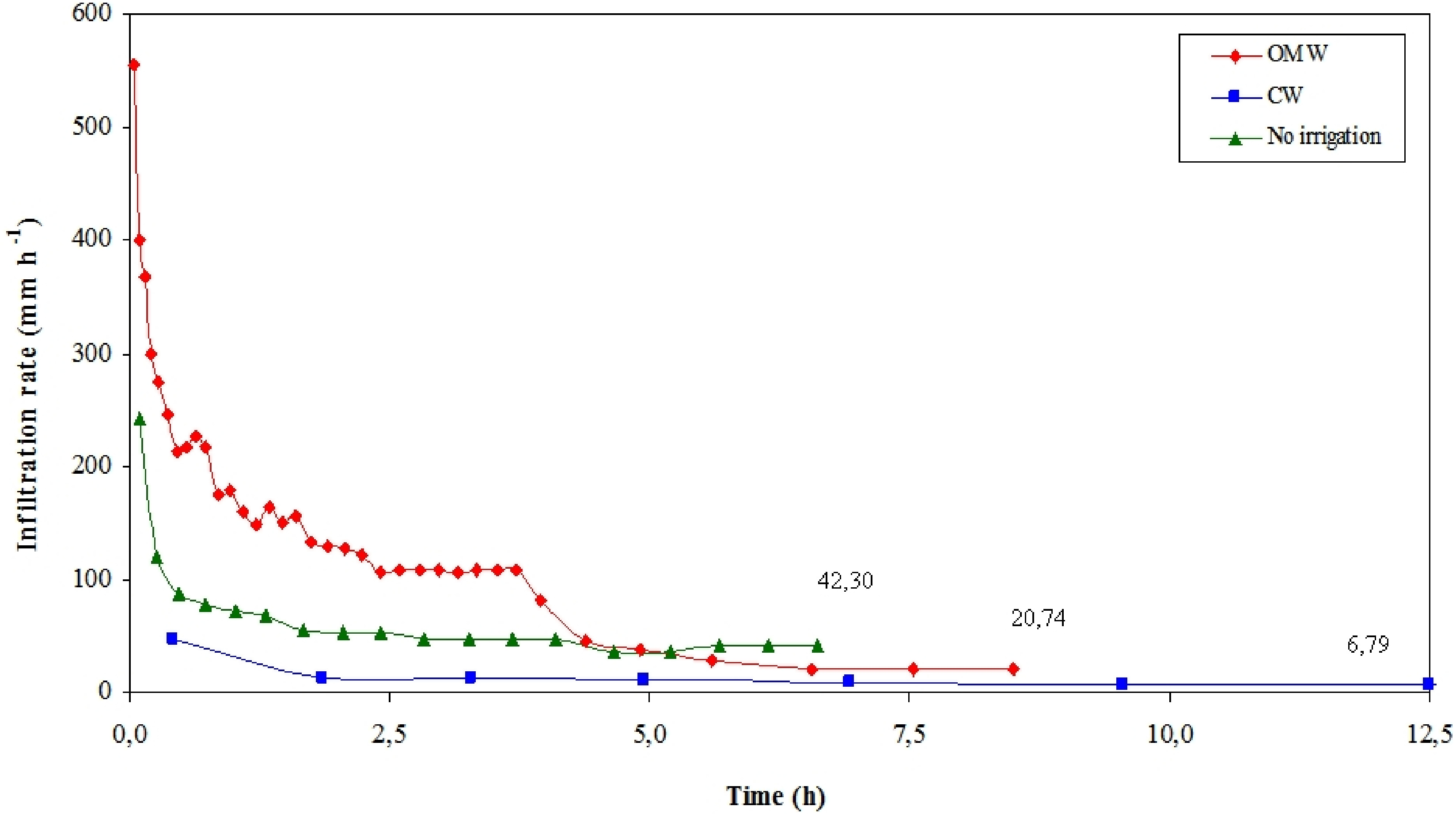
Figure 2 - Soil infiltration curves three weeks after treatments (points are averaged among replicates).

Measurements of soil infiltration rate by the double-cylinder infiltrometer until soil saturation. The last point of the curve is the steady-state infiltration rate.

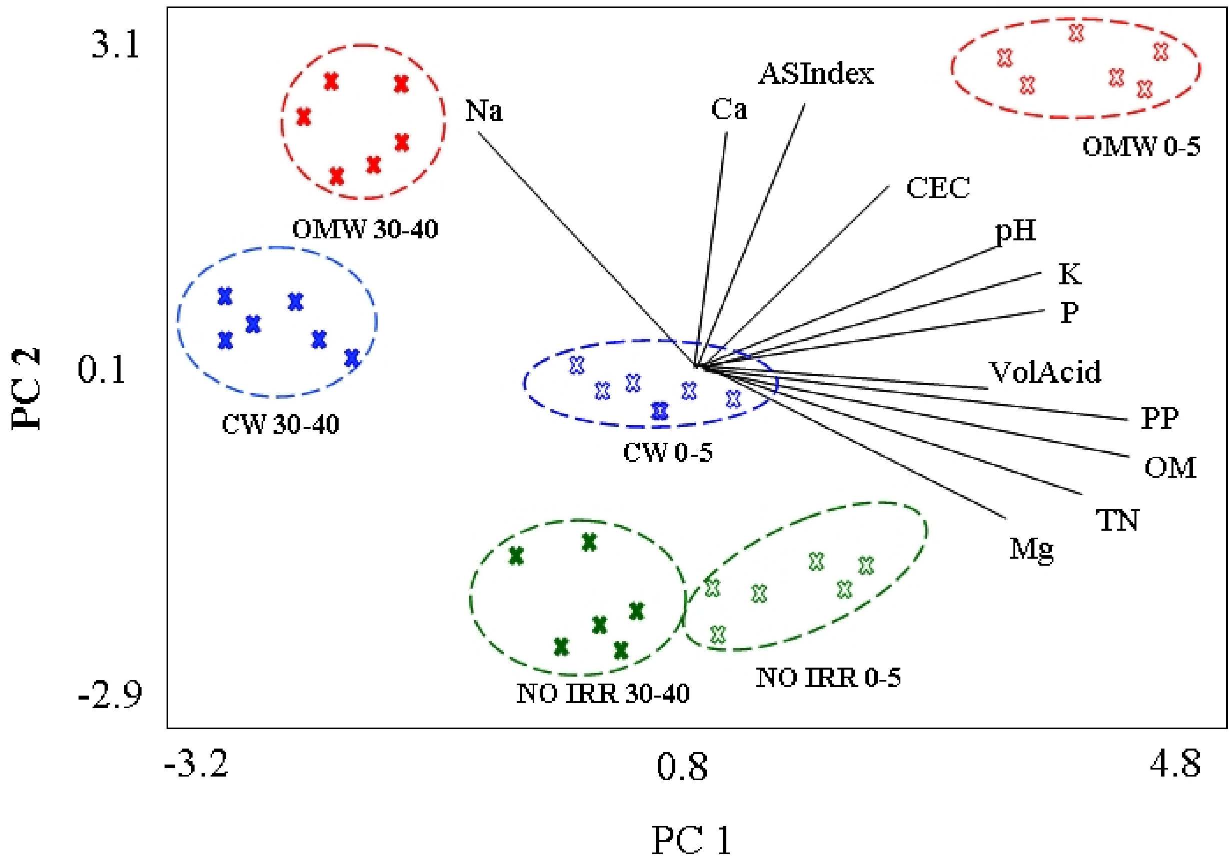
Figure 3 - Biplot of Principal Component Analysis applied to physico-chemical parameters of soils subject to treatments.

The first two principal components (PC1 and PC2) explain about 75% of the total variance of the original variables (47% for PC1 and 28% for PC2). Six clusters of scores (circled by a dashed line) on the two PCs are evident:: i) irrigation with OMW + 30-40 sampling depth (red full cross); ii) irrigation with OMW + 0-5 sampling depth (red empty cross); iii) irrigation with CW + 30-40 sampling depth (blue full cross); iv) irrigation with CW + 0-5 sampling depth (blue empty cross); v) no irrigation + 30-40 sampling depth (green full cross); and vi) no irrigation and 0-5 sampling depth (green empty cross).


LAMEZIA TERME
(a)
(b)
Olive grove (24 x 15 m)
Plot 1
Plot 2
Plot 3
(x 2 sampling depth)
Irrigation with OMW
Irrigation with CW
No irrigation (control)
Sampling point
Infiltration tests
(randomly distributed)



600
500
400
300
200
100
0
Infiltration rate (mm h -1)
0,0
2,5
5,0
7,5
10,0
12,5
Time (h)
OM W
CW
No irrigation
42,30
20,74
6,79


3.1
0.1
-2.9
PC 2
-3.2
0.8
4.8
PC 1
OMW 30-40
CW 30-40
CW 0-5
OMW 0-5
NO IRR 30-40
NO IRR 0-5
Na
Ca
ASIndex
CEC
pH
K
P
VolAcid
PP
OM
TN
Mg