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Evolution of several soil properties following amendment with olive mill wastewater

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ABSTRACT

Olive mill wastewater (OMW) is the main waste product of the olive oil industry and is characterized by high salinity and high organic matter content. Until today, in several olive oil-producing countries, untreated OMW is pumped into agricultural land with potential adverse effects on soil properties. The main objective of this study was to investigate the effects of OMW application on soil physicochemical proprieties. Three OMW levels (50, 100 and 200 m^3 ha^{-1} $year^{-1}$) were applied over eight successive years. Electrical conductivity, pH, total phosphorus and total nitrogen were studied at different soil depths. Results showed that OMW infiltration caused a modification of soil physicochemical characteristics. The most important effects on soil composition included a significant increase in P and N availability, which enhanced soil fertility in the OMW-treated soil.

Keywords: Agricultural reuse; Olive mill wastewater; Soil characteristics

1. Introduction

The Mediterranean region represents the most important olive-growing area in the world with around 30 million m^3 of OMW produced annually [1,2]. Tunisia is one of the largest olive oil producers in the world with an average annual production of 200,000 tons. This produces highly polluted wastewater and/or solid residue, seasonally between November and February, depending on the olive oil extraction process. The amount of OMW varies between 6×10^5 and 7×10^5 m^3 $year^{-1}$ [3] and its characteristics depend on the olive variety and ripeness, climate and soil conditions and the oil extraction method [2,3]. Olive oil production involves one of the following extraction processes: (i) press olive oil extraction, (ii) three-phase centrifugal olive oil extraction and (iii) two-phase centrifugal olive

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oil extraction [2]. The volume of OMW produced in traditional presses and in three-phase extraction systems amounts to about 600 and 1,000 L per ton of processed olives, respectively, while it is much lower in the two-phase process [4]. Moreover, OMW contains high concentrations of phenolic compounds that are phytotoxic and difficult to biodegrade [5]. Mekki et al. [6] detected phenolic compounds at a depth of 1.2 m four months after the last application of OMW, and a moderate phytotoxic residual phenolic fraction was extracted from the topsoil layer one year after OMW application. OMW has been shown to inhibit the arbuscular mycorrhizal fungal root colonization, which reduces the nutrient uptake of the olive trees [7]. OMW is becoming a serious environmental problem because of its high organic chemical oxygen demand (COD) concentration, phytotoxic properties and resistance to biodegradation due to the high polyphenol and organic content [8]. OMW disposal is a serious problem for producers, millers and the whole community due to its potential pollution risk [6,9,10]. To solve the problems related to the residue, different disposal methods based on evaporation ponds, thermal concentration, physicochemical and biological treatments have been proposed [11–14]. However, the direct application to agricultural soils as organic fertilizers is the most frequently used method nowadays [10,15]. The need for OMW disposal, on the one hand, and water scarcity and low soil fertility in olive-producing countries, on the other hand, have led to the fact that large amounts of OMW are used for irrigation and fertility purposes in Tunisia and other Mediterranean countries. OMW physicochemical composition and characteristics have been well documented [14,16]. Little is known, however, about the impact of OMW on the chemical, biochemical and biological soil properties [14,16,17].

The present work aimed at evaluating the occurrence of OMW amendments on several physicochemical and biochemical soil properties.

2. Materials and methods

2.1. Study sites and sampling

The experimental site was a field of olive trees (*Olea europaea*, L., variety Chemlali) located in ˝Chaâl" experimental station, in Sfax region, Tunisia (Fig. 1). The climate in this part of the country is typically semi-arid Mediterranean with an average annual rainfall of 210 mm. The field was divided into four plots, three of which were annually amended with OMW each January in one application (during 11 years). The three experimental plots T1, T2 and T3 have been amended with 50, 100 and 200 m^3 ha^{-1} of OMW (respectively) [6]. The plot T0 was not amended and served as control.

Soil samples were collected from each plot at a depth of 0–80 cm, using a soil auger. All soil samples were taken three months after OMW spreading. All the collected soil samples were air-dried, passed through a 2 mm sieve and then stored at 4°C prior to analysis.

2.2. OMW characterization

Fresh OMW was taken from a three-phase discontinuous extraction factory located in Chaâl, Sfax, southern Tunisia. This OMW was first recovered in a storage basin for one week, and then characterized before application.

Electrical conductivity (EC) and pH were measured using a conductivity metre and a pHmetre, respectively. The dry weight and moisture content were determined by weighing the sample before and after drying overnight at 105°C. Chemical oxygen demand was determined according to the Knechtel method [18]. Biochemical oxygen demand was measured using the respirometric method. Organic matter content was determined after heating the samples to 550°C in a laboratory furnace for 4 h and the phenol content was measured by the Folin–Ciocalteau method [19]. Total nitrogen was determined by the Kjeldahl method [20]; P was measured calorimetrically [21] and K, Na, Ca and Mg by atomic absorption spectrophotometry (HITACHI Model Z-6100).

2.3. Soil analysis

Soil analyses for pH, EC, N, K, P, Na and organic matter were performed three months after OMW application at four depths: 0–20, 20–40, 40–60 and 60–80 cm. Soil samples were air-dried, ground and sieved (2 mm). Soil pH and EC were determined using a pH metre (Model EA940, Orion, USA) and a conductivity meter (Model WTW LF 90), respectively. K, Na, Ca and Mg were determined by atomic absorption spectrophotometry (HITACHI Model Z-6100). Polyphenols in soil were monitored as follows: 50 g of soil was washed with 100 mL of hexane and then polyphenols were extracted with 250 mL of ethyl acetate and measured by the Folin–Ciocalteau method [19]. Total soil N was determined by the Kjeldahl method [20] and P by Olsen and Sommers method [21]. The soil organic matter was analysed by Walkley–Black method [22].

2.4. Phenolic compound extraction and determination

Polyphenolic compounds from the soil samples were extracted with ethyl-acetate, a highly selective

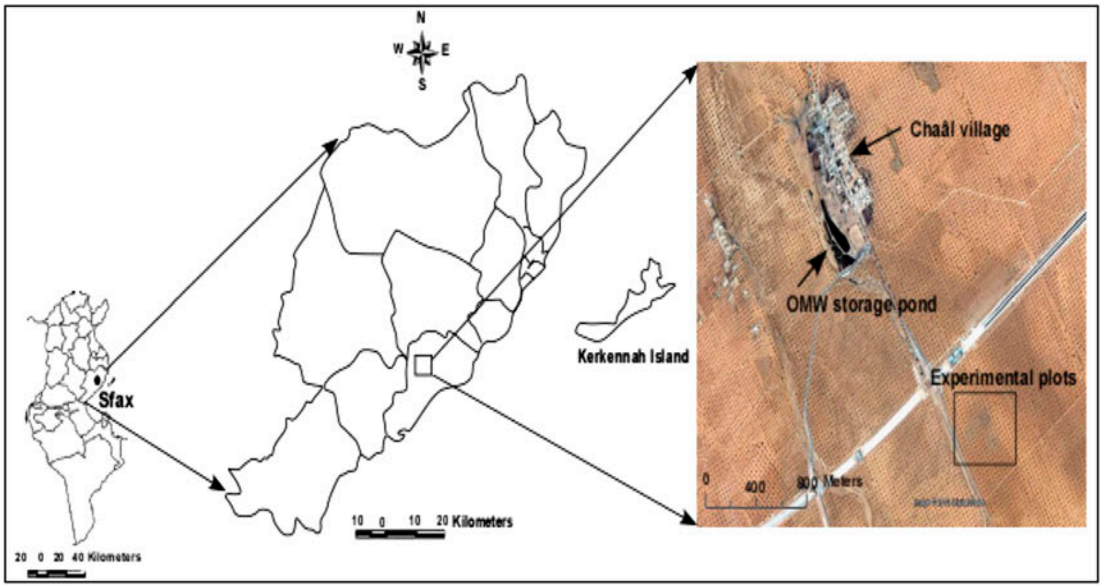

Fig. 1. Map of the experimental site located in Chaâl experimental station, in Sfax region, Tunisia.

solvent of low to medium molecular weight phenolic compounds. The soil polyphenols were monitored as follows: the samples were extracted with ethyl-acetate using a ratio of 5:1 (v/w). The collected organic fraction was then dried and evaporated under vacuum at 40˚C in a rotary evaporator. The dry residue was then re-dissolved in methanol. For the determination of total polyphenol contents, the Folin–Ciocalteau colorimetric method was used [19]. This involved the successive addition of 5 ml of sodium carbonate (6%) and 2.5 ml of Folin–Ciocalteau phenol reagent to a 50 ml sample. The solution was left in the dark for 20 min at 20˚C, after which the absorbance was measured at 750 nm, using gallic acid as a standard. Total polyphenolic values were expressed as gallic acid equivalents.

2.5. Statistical analysis

Statistical analyses were performed using the SPSS 17.0 Windows. The treatments' means were compared using Duncan's test calculated at 5% level. At least three replicates were used for each laboratory and field test.

3. Results and discussion

3.1. OMW characteristics

OMW characteristics depend on the olive variety and ripeness, climate and soil conditions and the oil extraction method. Selected physical and chemical characteristics of the OMW applied at the experimental sites are given in Table 1 and correspond to the mean values of three analyses. OMW was acidic at pH 4.63, with high organic load (COD 87 g L^{-1} and TOC 24 g L^{-1}) and high phenol content (4.2 g L^{-1}). OMW C/N ratio was unfavourable for the biodegradation and humification processes. OMW is characterized by a high concentration of several organic compounds, including sugar, tannin, pectin, lipids and phenolic substances [23–24], which are responsible for their high COD and BOD.

3.2. Soil characterization

The physicochemical characteristics of the soil of the study area are summarized in Table 2. The soil had an important content of active calcareous (4.3%) at the surface and was composed of sand (94.3% w/w), clay (4.7% w/w) and silt (1.3% w/w). It had an alkaline pH (7.88) and a weak electrical conductivity. The soil was very poor in nitrogen (0.16 g kg^{-1} dry soil) and organic matter (0.68% w/w). The level of phosphorous was 52.5 mg kg^{-1}.

3.3. Analytical results of soil profiles

As a vital component of soil fertility, the soil chemical property reflects its potential ability to provide nutrients for plants [25,26]. For soil profiles,

Table 1
Physico-chemical characteristics of olive mill wastewater used in ferti-irrigation

Characteristics	Data
pH (25 ℃)	4.63
Electrical conductivity (25℃) (dS m^{-1})	14.53
Chemical oxygen demand (g L^{-1})	87
Biochemical oxygen demand (g L^{-1})	40
COD/DBO5	2.2
Salinity (g L^{-1})	13.20
COT (g L^{-1})	24
Total solids (g L^{-1})	70.25
Mineral matter (g L^{-1})	13.2
Volatile solids (g L^{-1})	57.05
Total nitrogen (g L^{-1})	0.34
Carbon/nitrogen	117
P (g L^{-1})	0.19
Na^+ (g L^{-1})	1.4
Cl^- (g L-1)	3
SO_4^{2-} (g L^{-1})	1.58
K^+ (g L^{-1})	2.4
Ca^{2+} (g L^{-1})	0.38
Mg^{2+} (g L^{-1})	0.32
Fe^{2+} (mg L^{-1})	2
Total phenols (g L^{-1})	4.2

Table 2
The field experimented soil characterization

Properties	Soil layer (cm)	
	0–20	60–80
Particle size distribution		
Sand (%)	94.3	95.4
Silt (%)	1.3	3.5
Clay (%)	4.7	1.1
Physico-chemical characteristics		
pH	7.88	8.11
Electrical conductivity (μS cm^{-1})	595	433
CEC (ppm)	4.69	4.84
Organic matter (%)	0.18	0.18
Total nitrogen (mg kg^{-1})	161	178.5
P (mg kg^{-1})	52.45	53.85
$CaCO_3$ (%)	4.3	8.3

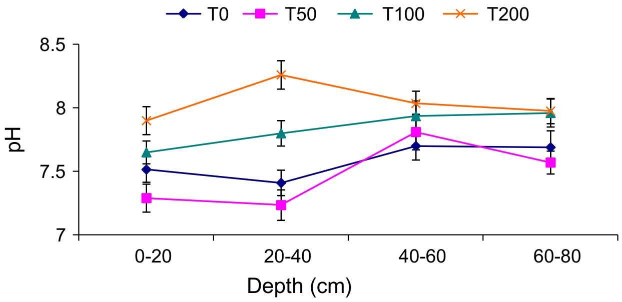

Fig. 2. Soil pH evolution according to amended OMW. Error bars represent ± one standard deviation from the mean, $n = 3$.

pH, electrical conductivity, organic matter and polyphenols contents were analysed three months after OMW spreading. The pH does vary according to depth. However, a significant increase in pH was observed when OMW spreading quantity increased (Fig. 2). The pH of the control plot was about 7.5 for each layer. Despite the acidic pH of OMW, a slight increase in pH values was observed for the soil amended with a dose of 100 and 200 m^3 ha^{-1}. The pH increase did not exceed 0.5 units for the soil treated with 200 m^3 ha^{-1} in relation to the control soil. As shown in Fig. 1, the pH of the soil amended with 50 m^3ha^-1 was not statistically different from the control soil. Mekki et al. [6] reported that pH does not vary according to depth. Mkhabela and Warman [27] suggested that the increase of pH may be due to the mineralization of carbon and the subsequent production of OH ions by ligand exchange, such as K^+, Ca^{2+} and Mg^{2+}, or to the Na brought by this waste which generates $NaCO_3$ of more alkaline hydrolysis than the $CaCO_3$.

Fig. 3 shows that electrical conductivity increased proportionally to the increase in the OMW quantity and decreased with depth in all analysed soils. While examining the layers 40–60 cm and 60–80 cm results, no significant differences in the EC values were noted for 100 and 200 m^3 ha^{-1} of OMW spreading rates. EC values in soils decreased in general with depth, indicating the infiltration of OMW from the surface to deeper soil horizons. In general, EC increase seems to

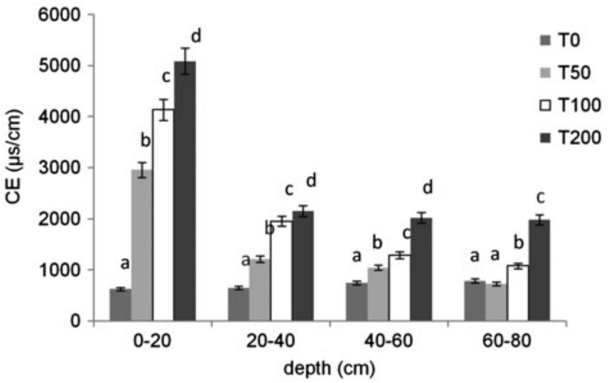

Fig. 3. Soil electrical conductivity evolution as a function of amended OMW. Means with different letters indicate a significant difference at $P \leqslant 0.05$ using Duncan multiple range test.

be irreversible, especially in the upper soil layers when excessive OMW rates are applied [28–30]. Increase in soil EC is mainly due to ionic species, namely potassium, chloride, sulphate, ammonium and nitrates, present either in OMW or generated through waste mineralization and transformation [10].

This EC increase could result from the main ionic species: sodium, chloride and sulphate (Table 1). However, the decrease of EC on the lower layers could be related to the phenomena of adsorption and evapotranspiration which concentrate on the upper layer. The phenomena are related to the experimental period when soil samples were taken after the hot season.

Soil organic carbon content, which is mainly influenced by climate and vegetation, is the result of the equilibrium between the inputting organic matter and the departing organic matter mainly caused by soil microbial decomposition [31,32]. Soil organic matter is one of the most important components of soil fertility [33]. The analysis of organic matter content in the various soil layers (Fig. 4) showed that OMW induced an increase in the organic matter content in higher soil layers with a slight migration towards the lower layers. The content of organic matter at the plots T100 and T200 was very high in the upper soil layers (0–20 cm) but decreased sharply with depth. The chemical soil properties contributed by soil organic matter include the mineralization of nutrients and their availability to plants, cation exchange capacity and binding of heavy metals and pesticides. In fact, for plants soil, OM is the basic resource of several elements. Normally, 95% of N and S reside in organic matter [34]. The proportion of P associated with soil OM is variable. According to Sanchez [35], 60–80% of soil P is of organic origin, while Stevenson [34] calculates its part between 20 and 80%. Thus, a fall in organic matter represents a serious suppression in nutrient availability [36]. In addition to being a direct source of essential plant nutrients, OM also indirectly influences nutrient availability, e.g. by an increased availability of phosphates [34], an acceleration in availability of Fe and Mn on submergence [37], an increased availability of trace elements through complexation by organic ligands or a decrease in toxicity, a fall in ammonia volatilization [38] and a buffering of soil pH, more specifically of poorly buffered low CEC soils [39]. Diminishing OM led to a significant decline in the availability of Zn, Cu, Mn and Fe [40].

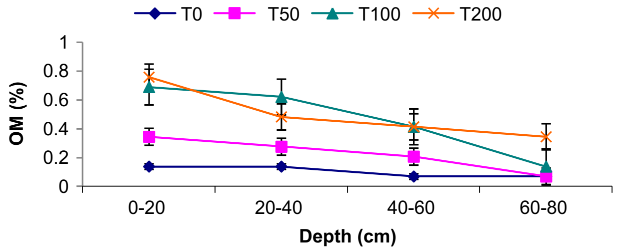

Fig. 4. Soil organic matter evolution as a function of amended OMW. Error bars represent ± one standard deviation from the mean, $n = 3$.

Elements of N and P are two types of mineral elements that are absorbed in large amounts by a plant from the soil. N and P have been viewed as the two most important elements that limit plant growth in most plants [41,42]. The mean total nitrogen content in surface soils at T50, T100 and T200 plots (200, 250 and 300 mg g^{-1}) was relatively higher than the values recorded in the respective control site T0 (160 mg g^{-1}) (Fig. 5). N is the most important micro-element in the soil for plant development [43]. On the other hand, excess of N, caused by fertilization or waste disposal, may result in water eutrophication or cause health problems to humans [43].

Fig. 6 also shows that the available phosphorus content increased after a dose was applied. The spreading of OMW on soil involved the enrichment of the high soil layers by P with a small quantity which migrated towards the lower layers even with a higher

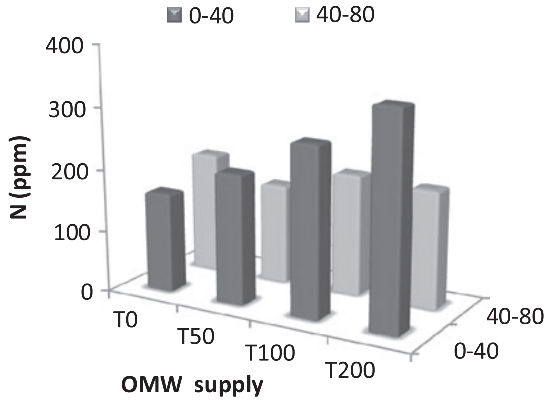

Fig. 5. Effect of OMW application on soil total nitrogen content at two soil depths (0–40 and 40–80 cm).

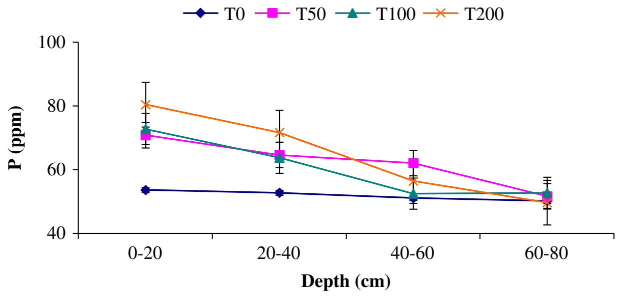

Fig. 6. Effect of OMW application on soil phosphorus content at four soil depths (0–20, 20–40, 40–60 and 60–80 cm). Error bars represent ± one standard deviation from the mean, $n = 3$.

Table 3
Amount of soil polyphenol obtained from the difference control after OMW spreading of different doses: 50; 100 and 200 m^3 ha^{-1} in the two soil layers (0–40 and 40–80 cm). Means with different letters (a,b and c) indicate a significant difference at $P \leqslant 0.05$ using Duncan multiple range test

OMW supply (m^3 ha^{-1})	50	100	200
Depth (cm)			
0–40 cm	1440^a	1527^a	1746^b
40–80 cm	174^a	436^b	480^b

dose (200 m^3 ha^{-1}). Carreira et al. [44] indicated that paedogenic $CaCO_3$ is the primary geochemical agent in arid ecosystems capable of reducing leaching losses of P, through secondary precipitation of Ca-P minerals and/or strong sorption reaction of P with $CaCO_3$, which has broadened ecosystem implications for the retention of P, within both the soil profile and the landscape.

3.4. Phenolic compounds dynamics

This part of the study investigated the dynamics of the phenolic compounds in a very porous and permeable sandy soil (Table 2) irrigated by increasing doses of OMW 50,100 and 200 m^3 ha^{-1} $year^{-1}$ and traced the change of the phenolic compounds' content according to depth. Table 3 shows a significant increase in phenolic compounds' concentration with the increase of OMW dose in the upper ground level (0–40 cm) compared to the control. However, in the depth (40–80 cm), the increase in the concentration of phenolic compounds compared to the control was lower than that of the upper ground level. On the other hand, for the same depth of the soil, the increase in the phenolic compounds' concentration was not proportional to the increase in OMW doses 50; 100; and 200 m^3 ha^{-1}. Statistical analysis indicates a significant difference with doses 50,100 m^3 ha^{-1} of OMW. Therefore, 100 m^3 ha^{-1} and 200 $m^3 ha^{-1}$ doses of OMW indicate no significant difference.

Besides, Table 3 shows that polyphenolic compounds' content decreased with depth, within the successive investigated layers (0–80 cm); however, it remained high compared to control. This could be due to their decomposition or incorporation into the humic fraction of the organic matter present in the soil [45,46]. Soil biotic and abiotic components exert relevant capabilities of OMW phenolics degradation [47]. El Hassani et al. [48] reported that the abundance of total microflora in the soil was enhanced after OMW application, while no correlation was found between soil phenolics and soil microflora. Furthermore, Di Serio et al. [49] reported that OMW application in the soil increased the respiration activity with respect to the untreated soil, which is highly correlated with the organic matter decomposition in the soil. Higher respiration activity indicates the ability of the microorganisms to utilize and decompose variable organic substrates introduced with OMW.

4. Conclusion

Olive mill wastewater (OMW) amendment seems to affect the structure and composition of the soil. OMW spreading leads to soil organic matter content increase in accordance with the quantity applied. Results indicated that OMW application increased the soil fertility, offering the opportunity to recycle the various compounds. Because of the high amounts of organic matter and macronutrients (especially total nitrogen and phosphorus), OMW could be considered as a useful, low-cost amendment and fertilizer. In contrast, the electrical conductivity of the soil reached the salinization threshold. However, this practice should take into account the cumulative effect of soil salinization, which would little by little transform the soil into an unproductive one.

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