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Effects of Olive Mill Wastewater on Soil Nutrients Availability

Leïla Chaari[1]*, Nada Elloumi [2], Salma mseddi[1], Kamel Gargouri[3], Béchir Bourouina[3], Taher Mechichi[4], Monem Kallel [1]

[1] University of Sfax, Laboratory of Water, Energy and Environment, National School of Engineers of Sfax (ENIS), Sfax, Tunisia
[2] University of Sfax, Laboratory of Water, Energy and Environment, High Institute of Biotechnology of Sfax (ISBS), Sfax, Tunisia
[3] Laboratory of Soil Sciences, Water and Environment, Olive Tree Institute of Sfax(IO), Tunisia
[4] Laboratory of Enzyme Engineering and Microbiology, National School of Engineers of Sfax (ENIS), Sfax, Tunisia
*Corresponding author: Leïla Chaari

Abstract

Olive mill wastewater (OMW) is one of complex wastewater generated by the olive oil extraction process. It is characterized by high values of COD, BOD and phytotoxic levels of polyphenols, but also by a high amount of organic compounds and plant mineral nutrients. Therefore, OMW field spreading may represent a low cost contribution to soil amendment. Olive mill wastewater (OMW) spraying effects onto soil physico-chemicals characteristics were investigated. Three OMW doses 50, 100 and 200 m^3 ha^{-1} $year^{-1}$ were applied for ten successive years on sandy soil. The findings showed that the pH of the soil, electrical conductivity and organic matter, total nitrogen, phosphorus, sodium, and potassium soil contents increased with increased OMW supply. While, no variations were recorded in calcium and magnesium on soil contents.

Keywords: Olive mill wastewater (OMW); spreading; soil layers; available nutrients; phenolic compounds.

Introduction

The olive oil produced in the world is mainly from the Mediterranean countries, with Tunisia as one of the five major producers. Olive oil produces a highly polluted wastewater and it's depending on many factors, such as olives type and maturity, climatic conditions and region of origin, cultivation methods, and technology used for the extraction process. Olive mill wastewater (OMW) has been a critical environmental problem concern for the most olive oil producing countries. It is characterised by large volumes, salinity, low pH, high organic load and amount of toxical-phytotoxical compounds, such as polyphenol (Di Bene et al., 2013; Chaari et al., 2013).

Different disposal methods based on evaporation ponds, thermal concentration, physico-chemical and biological treatments as well as direct application to agricultural soils as organic fertilizers have been proposed (Rozzi and Malpei,1996; Kallel et al., 2009; Belaid et al., 2013). Some characteristics of OMW are favourable for agriculture, since this effluent is rich in organic matter, nitrogen, phosphorous, potassium and magnesium (Mechri et al., 2011; Chaari et al., 2013). So soils in semi arid areas have a very low nutrient availability and low organic matter content (Garcıa et al., 1994). OMW contains an important level of organic matter, considerable quantities of nutrients and are a non negligible water source (Ammar et al., 2005; Mechri et al., 2008). Moreover, it includes various simple and complex phenolic compounds, generating antimicrobial and phytotoxic effects (Hachicha et al., 2009). Although, its polyphenolic fraction was gradually degraded with time and partially transformed in to humic substances. Thus, polyphenol degradation and incorporation into the soil humic fraction depend on environmental conditions (Sierra et al., 2007). Several papers showed adverse effects on the direct application of fresh OMW (Azbar et al., 2004) such as soil salinization (Paredes et al., 1987; Abdullah and Khalid, 2007), phytotoxicity (Mekki et al., 2006; Saadi et al., 2007) and groundwater quality degradation (Saadi et al., 2007). Mekki et al. (2006) have reported that 50 m^3/ha/yr OMW application rate significantly decreasing the risk of groundwater contamination by avoiding the accumulation of high BOD load in the soil. Altieri et al. (2008) reported that OMW spreading on top soil may have beneficial effects such as nutrient availability for plant growth. Chartzoulakis et al. (2010) showed that after 3 years of raw OMW application there were no significant differences in pH, EC, P, Na and organic matter between control and OMW-treated soils. Existing data on effects of OMW on soil properties is several cases contradictory. However, the direct application to agricultural soils as organic fertilizers is the most frequently used method nowadays (Mekki et al., 2006; Ben-Rouina et al., 1999) and also using OMW for irrigation offsets the water scarcity and low soil fertility in the Mediterranean region.

The objective of the present investigation was to study the impact of OMW spreading on several physico-chemical soil properties and soil-phenolic compound evolution at the different soil-layers of designated olive tree test-site.

Materials and methods

Field investigation

The experimental site was a field located in "Chaâl" experimental station, at 60 Km South-West in Sfax region (Tunisia, latitude North 34° 3', longitude East 10° 20'). The climate of the region is typical Mediterranean, semiarid to arid, with an average rainfall of 210 mm year[1].

The olive-trees field was divided into four plots (T0, T50, T100 and T200). The latter three were annually spreading with the same annual dose of raw OMW each January (during 11 years). The experimental plots T50, T100 and T200 have been respectively spread with 50, 100, and 200 m^3 ha^{-1} of OMW (Mekki et al., 2007). The plot T0 was served as control.

Soil samples were collected from each plot at different layers 0-20, 20-40, 40-60 and 60-80 cm, using a soil auger. All samples were taken three months after OMW application, soil samples were collected, air dried, passed through a 2 mm sieve and then stored at 4 °C prior to analysis.

OMW applied characterization

Fresh OMW was taken from evaporation ponds on the extraction factory located in Chaâl and then was characterized before application. Electrical conductivity (EC) was measured using a conductivity meter and pH using pH-meter. The chemical oxygen demand and Biochemical oxygen demand were determined according AFNOR T 90-101 and the respirometric method respectively. Total phosphorus P was measured calorimetrically (Olsen and Sommers, 1982) and total nitrogen was determined as described on Kjeldhal method. The organic matter was measured after incineration samples at 550°C for 4 h. Total phenolic compounds was determined using the Folin–Ciocalteau method (Box, 1983) and K^+, Na^+, Ca^{2+} and Mg^{2+} by atomic absorption spectrophotometry (Thermo Fisher Scientific ICE 3000).

Soil analysis

Soil analyses for pH, EC, Na, K, Ca, Mg and organic matter (OM) were performed three months after OMW application at four depths: 0-20; 20-40; 40-60 and 60-80 cm. Soil EC and pH were measured by a conductivity meter (Model WTW LF 90) and pH meter (Model EA940, Orion, USA), respectively. Walkley-Black method was used for soil organic matter analyse. K^+, Na^+, Ca^{2+} and Mg^{2+} were extracted by ammonium acetate and determined by atomic absorption spectrophotometry (Thermo Fisher Scientific ICE3000).

Sodium adsorption ratio (SAR) was determined from Na^+ and Ca^{2+} plus Mg^{2+} in the soil solution (Sumner et al., 1998). In order to obtain the Sodium adsorption ratio (SAR), the following formula was used:

$$SAR= [Na^+]/ (([Ca^{2+}] + [Mg^{2+}])/2)^{1/2} \quad (1)$$

Polyphenol extraction and quantification

Polyphenols were extracted with using ratio 1:5 (v/w) with methanol from each sample soil (Avallone et al., 1997) and was measured by the Folin–Ciocalteau method (Box, 1983).

Results and Discussion

Characteristics of the soil and OMW used for irrigation

The soil texture characteristic is a sandy soil (clay 4.7%, silt 1.3%, sand 94%), with low OM and calcium carbonate contents (Table 1). It was moderately alkaline (pH = 7.5) and classified as a non saline soil (EC< 4 dS m^{-1}). The cation exchange capacity (CEC) was 6236 ppm. This kind of soil is characterized by very low fertility (0<CEC<10 mg/100g) and nutrients contents.

Table 2 shows the measured physicochemical properties of olive mill waste water. The OMW was acidic pH 4.63, with high pollution load composed of organic matter (116.3 g L^{-1}) and mineral matter (13.2 g L^{-1}). The analyses performed on this effluent showed that total organic carbon (TOC) and chemical oxygen demand (COD) values exceed the authorised standards limits and were 24 and 87 g/L respectively. Assay of polyphenols leads to an estimated value of around 4.2 g/L thought to be the origin of OMW toxicity. Belaid et al. (2013) showed that OMW-analysed by GC/MS led to identify carboxylic acids, fatty acids and 18 phenolic monomers among them neutral phenolic compounds and phenolic acids.

OMW effect on pH, EC and organic matter content of soil

The pH variations of the OMW amended soil at the different soil layers after OMW treatments are presented in Table 3. The results showed that the pH of the different soil layers varied from 7.4 to 8.3. During the OMW treatment process, the soil pH at surface horizons (0–20 cm), particularly those of the soil samples treated with 200 m^3 ha^{-1}, were noted to increase in comparison to the control. 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. Mekki et al. (2007) reported that pH does not vary according to the depth. Magdich et al. (2013) reported that the soil pH at surface horizons treated with OMW, were noted to decrease in comparison to the control, which could presumably be attributed to the acidic nature of OMW. Mkhabela and Warman (2005) reported that the increase of pH may be due to the mineralization of carbon which product 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 $CaCO_3$.

In this case, electrical conductivity (EC) was significantly affected by the application of OMW, showing increases after spreading in the treated soils compared with the controls. Nevertheless, EC values remained below the salinity threshold (4000 µS cm^{-1}), except for soil samples treated with 100 and 200 m^3 ha^{-1} in the upper layer. These results were consistent with previous works, reporting EC increases during the irrigation times (Chartzoulakis et al., 2010; Kavvadias et al., 2010; Moraetis et al., 2011; Di Bene et al., 2013).

As a consequence of irrigation with OMW, the OM content in the topsoil increased from 0.18% in the control to 0.4, 0.7 and 0.8 % after 50, 100 and 200 m^3 ha^{-1} of OMW application, respectively (Table 3). Moreover, the soil surface horizons (0–20 cm) were noted to exhibit the highest levels of soil organic matter content. The most important difference was particularly observed between the 200 m^3 ha^{-1} treated soil and non treated soil. Furthermore, for the treatment using the highest amount of OMW, the organic matter content recorded in the surface horizon reached 4 folds (0.76%) as compared to the control (0.18%).

OMW effect on phosphorus and nitrogen contents of soil

An increase in total N following irrigation with OMW was also been observed (Figure1). Furthermore, for the treatment using the highest amount of OMW, the total N content recorded in the surface horizon reached 2.2 folds (320 ppm) as compared to the control (150 ppm). Several studies showed an increase in organic matter content, total N and C/N ratio following irrigation with OMW and may have a beneficial effect on soil fertility (Mekki et al., 2006; Brunetti et al., 2007; Sierra et al., 2007; Mechri et al., 2008).

As regards phosphorus, results revealed a significant impact of the OMW disposal (Figure2). The application of OMW was also noted to enhance the total phosphorus content in the soil. The highest levels of P content were recorded at 0–20 cm depths for all the treatments. Magdich et al. (2013) reported that no improvement in soil P content was observed after 3 years of treatment with OMW.

Soil K, Ca and Mg contents progress

The highest increase of K content was recorded at the upper layers of the soil (0–20 cm) (Table 4). In fact, the most important level of soil K content (1250 mg kg^{-1}) was registered at the surface layer (0–20 cm) of the soil treated with 200 m^3 OMW ha^{-1} $year^{-1}$. This level was almost 10 times higher than that in the control. The lowest soil K content values were found at the deeper layers of soil with all the different assayed treatment of OMW spreading. Di Bene et al. (2013) reported that K_{exch} was significantly affected by the OMW treatment at both spreading sites and, just after OMW disposal. According to this author K_{exch} values were found to be four to 10 fold higher than the observed ones in the control. Magdich et al. (2013) has reported a significant increase of soil K concentration with a value of around (985 ppm) at the surface layer (0–20 cm) of the soil treated with dose 200 m^3/ha/ yr of OMW. Accordingly, this level was almost 6 times higher than that in the control. These results provide support for the proposals in the literature the candidacy of OMW for application as an alternative K fertilizer (Montemurro et al., 2004; Di Serio et al., 2008).

As far as the soil calcium and magnesium contents were concerned, no significant difference were noted between the treatments for the different levels of OMW soil amendment (Table 4). Chartzoulakis et al. (2010) reported that the only significant changes in soil composition observed in the soil treated with OMW were the increase of soil K and total phenols, in accordance to previous reports (Paredes et al., 1987; Levi-Minzi et al., 1992; Mechri et al., 2008). The increase in soil K can improve soil fertility and reduce the use of chemical fertilizers. Sopher and Baird (1982) reported that the ideal cation ratios recommended for plant use were: Ca/Mg, 6.5/1, Ca/K 13/1, and Mg/K, 2/1. The ratios Ca/K and Mg/K after OMW decreased as compared to control. A significant factor affecting plant mineral nutrition is the Ca and Mg ratio (Ca/Mg) in the soil. The most optimal Ca/Mg ratio in soils for plant nutrition is 5–8/1(Rinkis and Nollendorf , 1982). The average Ca/Mg ratio found in our study was higher, generally between 17/1 and 10/1, and therefore unfavourable for Ca uptake from the soil. This ratio is 17/1for untreated soil and decrease in treated soil with 200 m^3 OMW ha/ yr to 10/ 1.

Sodium content and SAR value progress

The potential impact of irrigation water quality on soil structure can be evaluated using sodium content and sodium absorption ratio (SAR) values, as shown in Figure 3. Compared to the control, significant increments were observed in terms of sodium absorption ratio (SAR) value and the sodium content distributions in the different soil layers under the different rates of OMW spreading. The highest increase of sodium content and SAR values was recorded at the upper layers of the soil (0–20 and 20–40 cm).The sodium concentration at the upper layer (0–20 cm) of soil treated with the highest rate of OMW was almost two folds higher than that of the control (Table 3). Soils accumulate sodium is in direct relation to the sodium absorption ratio (SAR) of applied OMW. The greater amount of salts in OMW supplied to the soil may have increased Na concentrations in the soil (Figure 3). However, the SAR values were far below the limit

(> 15) established to define saline-sodic soils (Mahdy, 2011). If irrigation water with a high SAR is applied to a soil for years, the sodium in the water can displace the calcium and magnesium in the soil. This will cause a decrease in the ability of the soil to form stable aggregates and a loss of soil structure. Mahmoud et al. (2010) reported that high concentrations of K and Na in the applied OMW probably led to an increase in exchangeable sodium percentage (ESP) and a subsequent degradation of soil structure. Micheal et al. (2008) reported that oil and grease in wastewater used for irrigation can accumulate in the soil and may lead to a significant reduction in the soil's ability to transmit water.

Polyphenolic compound content in the soil

The high content of polyphenols in the OMW strongly affected their occurrence in the soil. The polyphenolic compound content in the soil showed an increase with the increase of OMW spraying.

We noted also a decrease of polyphenolic content with depth (Table 5), within the successive investigated layers (0 to 80 cm). Jarboui et al. (2008) showed that in the upper soil layer, the polyphenol cumulative effect was influenced by soil structure and the substrate solubility.

Conclusion

The results from this study showed that the fertilization potential of OMW is considerable especially for N, P and K. The total NPK fertilization with 200 m^3 OMW ha^{-1} $year^{-1}$resulted in 2 times more nitrogen and phosphorus and 10 times more potassium than control. Electrical conductivity in the soil remained below the salinization threshold therefore no impact was observed on the soil quality. However, the addition of 200 m^3/ha could affect the salinity of the soil after long-term applications of OMW.

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Table 1. Characterization of Chaal soil sample.

Properties	Soil layer (cm)	
	0-20	20-80
Sand (%)	94.3	95.4
Silt (%)	1.3	3.5
Clay (%)	4.7	1.1
pH	7.5	7.4
EC (µS cm^{-1})	631	653
Ca CO_3 (%)	4.3	8.3
Organic Matter (%)	0.14	0.14
Ca^{2+} (mg/kg)	5260	5340
Mg^{2+} (mg/kg)	340	380
K^{+} (mg/kg)	144	136
Na^{+}(mg/kg)	59	90
CEC (mg/kg)	6236	5979.4

Table 2. Physico-chemical properties of OMW effluent spread

Characteristics	Average value
pH	4.63± 0.48
EC (dS cm^{-1})	14.53±0.20
TOC(g L^{-1})	24± 0.93
COD (g L^{-1})	87±1.09
OM (g L^{-1})	116.3±4.1
Minerals matters (g L^{-1})	13.2± 0.28
Nitrogen (g L^{-1})	0.34± 0.25
Carbon/Nitrogen	117
P (g L^{-1})	0.19±0.049
Na^{+} (g L^{-1})	1.4± 0.05
K^{+} (g L^{-1})	4.3± 0.05
Ca^{2+} (g L^{-1})	0.38± 0.05
Mg^{2+} (g L^{-1})	0.32± 0.05
Ca /Mg	1.18
Total phenols	4.2± 0.49

Table 3. Evolution of soil pH, electrical conductivity (µS cm^{-1}) and organic matter content (%) at the different layers in the experimented field.

	Layers (cm)	Doses (m^3. ha^{-1}year^{-1})			
		0	50	100	200
pH	0-20	7.5	7.3	7.6	7.9
	20-40	7.4	7.2	7.8	8.3
	40-60	7.7	7.8	8	8
	60-80	7.7	7.6	8	8
EC (µS/cm)	0-20	631	2955	4135	5085
	20-40	653	1217	1960	2155
	40-60	749	1049	1292	2025
	60-80	788	730	1082	1985
OM(%)	0-20	0.18	0.35	0.69	0.76
	20-40	0.18	0.28	0.62	0.65
	40-60	0.07	0.20	0.41	0.45
	60-80	0.07	0.07	0.14	0.34

Table 4. Evolution of soil total potassium, calcium and magnesium contents (mg/kg) at the different layers in the experimented field.

Treatment	Depth	K	Ca	Mg	Ca/Mg	Ca/K	Mg/K
Control	0-20	144	5260	340	15.47	36.53	2.36
	20-40	136	5340	380	14.05	39.26	2.79
	40-60	101	5320	420	12.67	52.67	4.16
	60-80	101	5260	400	13.15	52.08	3.96
T50	0-20	550	5200	300	17.33	9.45	0.55
	20-40	172	5180	340	15.24	30.12	1.98
	40-60	89	5120	320	16.00	57.53	3.60
	60-80	79	4940	340	14.53	62.53	4.30
T100	0-20	850	5220	320	16.31	6.14	0.38
	20-40	400	5160	360	14.33	12.90	0.90
	40-60	142	5120	400	12.80	36.06	2.82
	60-80	99	5000	340	14.71	50.51	3.43
T200	0-20	1250	4900	460	10.65	3.92	0.37
	20-40	700	5060	440	11.50	7.23	0.63
	40-60	550	5020	360	13.94	9.13	0.65
	60-80	181	4860	300	16.20	26.85	1.66

Table 5. Amount of soil polyphenols after OMW spreading at two soil depths (0–40 and 40–80 cm). Means with letters a b c and d indicate a significant difference at P ≤ 0.05.

	OMW applied ($m^3 ha^{-1}$)			
Layers (cm)	0	50	100	200
0-40	1571^{a}	2967^{b}	3403^{c}	3883^{d}
40-80	1876^{a}	2138^{b}	3010^{c}	3622^{d}

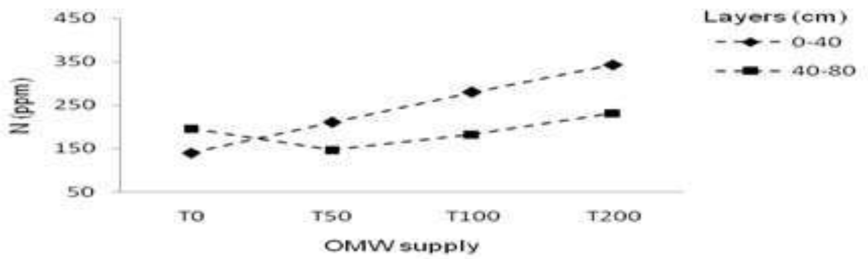

Figure 1. Effect of OMW application on soil total nitrogen content at two soil depths (0–40 and 40–80 cm).

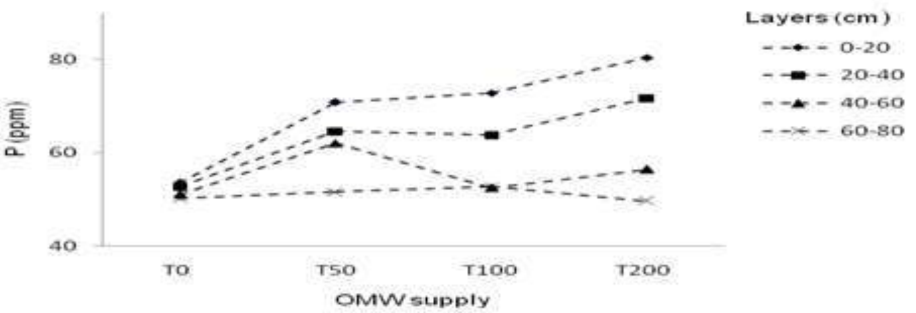

Figure 2. Effect of OMW application on soil phosphorus content at four soil depths (0–20, 20–40, 40–60 and 60-80 cm).

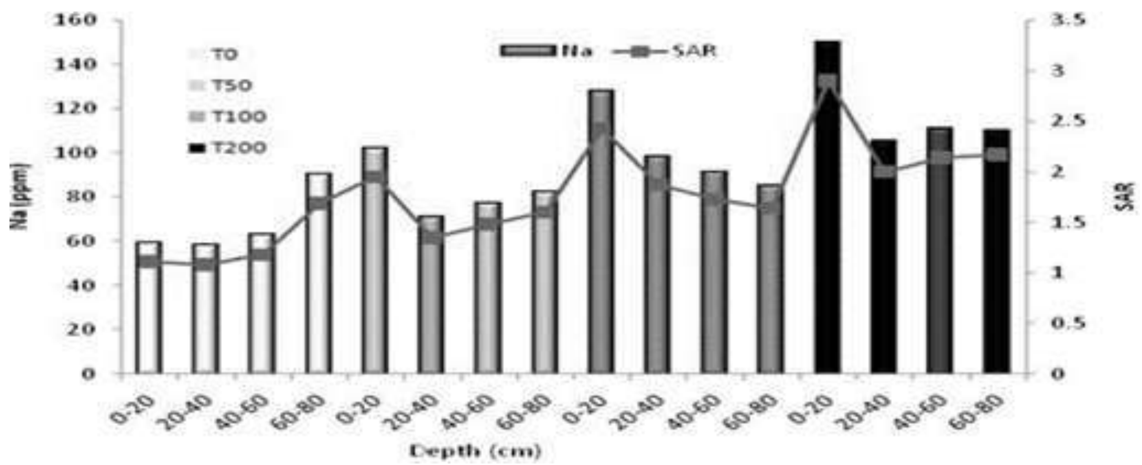

Figure 3. Evolution of sodium absorption ratio (SAR) and sodium content (ppm) at the different layers in the experimented field.