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Effect of olive mill wastewater spreading on the physicochemical characteristics of soil

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ABSTRACT

Olive mill wastewater (OMW) is the liquid by-product obtained from olive processing to extract virgin olive oil. Due to its acidic pH and high amounts in organic matter and phenols, OMW is very difficult to further purify. A solution would be to spread it on the soil. Hence, the objective of this study was to explore the effects of different OMW amounts on chemical characteristics of the soil cultivated with vineyard (cv. Italia), at different depths (10–30 cm and 30–60 cm).

Our results show that after 2 months of spreading follow up, during 2 consecutive years (2005/2006 and 2006/2007), the upper soil layer (10–30 *cm*) of land plots irrigated by OMW became fertile; with an average ratio *N.P.K* of respectively, 1.54; 1.95 and 2. This study also showed the absence of risk of soil filling by the suspended matter and the residual oil brought by OMW. In the same way, the pH and the electric conductivity (*EC*) of the soil remain practically unchanged. The concentration of OMW in calcium as well as the high content of soil limestone favoured the precipitation of limestone ($CaCO_3$), especially in the upper soil layer (10–30 cm). An increase in soil organic matter and phenols, due to spreading with OMW, was found as estimated by direct cell counting.

***Keywords*:** Olive mill wastewater (OMW); Fertilizing; Spreading; Clay soil; Total phenols

1. Introduction

Olive processing by mechanical means allows to obtain virgin olive oil but also by-products, such as olive pomace and olive mill wastewater (OMW). The pomace is valued economically because it is generally destined to the industry for extraction of pomace oil by solvent (hexan), while the elimination of OMW represents one of the main environmental problems related to the olive oil industry in Mediterranean countries.

The OMW composition is highly variable and depends, in particular, on variety, ripeness degree and type of the oil extraction technology (traditional or continuous systems). Generally, OMW has high values of biological oxygen demand (BOD) and chemical oxygen demand (COD), high contents of organic matter, suspended matter (Andrich *et al.* 1986; Di Giovacchino *et al*. 1988; Proietti *et al.* 1995) [1–3], inhibiting substances (lipidic and phenolic compounds), and a medium–high content of the mineral fraction, in particular of potassium, phosphorus and calcium. OMW is often rejected anarchically into rivers, spread in an uncontrolled way on agricultural soil or stored in basins,

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thus exposing the systems water-soil-plant to an unavoidable pollution. Physicochemical and biological treatments of OMW (Hamdi and Ellouz 1993; Flouri *et al*. 1996; Chimi 1997; Sayadi *et al*. 2000; Zenjari 2000; Sabbah *et al*. 2004; Di Giovacchino *et al*. 2005) [4–10] which consisted in reducing their impact on the water resources remain still insufficient and expensive. There are few studies on impact of OMW on soil physicochemical and microbial composition (Marsilio *et al.* 1990; Cicolani *et al*. 1992; Rhouma *et al*. 2008) [11–13]. On the other hand, many studies have been published on the opportunity to utilize OMW in the Mediterranean countries, both by spreading on agricultural land and by its purification with different techniques (Amirante and Di Renzo 1990; Brunetti *et al.* 1991) [14–15], but the results obtained were not easily practicable. Other authors pointed out the beneficial effects of OMW spreading, especially its positive action on soil fertility (Morisot 1979; Janer Del Valle 1980) [16–17]. Recent papers were published about the effects of OMW spreading on the soil cultivated with grapevine (Di Giovacchino *et al*. 2001; Rinaldi *et al.* 2002; Marsilio *et al*. 2006) [18–20]. Furthermore, the chemical characteristics of the soil treated with different amounts of OMW (Levi-Minzi *et al.* 1992; Saviozzi *et al.* 1993; Proietti *et al*. 1995) [3,21,22], the structure of the soil (Favi *et al*. 1993) [23] were also studied. The results indicated that a controlled spreading of OMW increased the soil fertility, offering the opportunity to recycle the various compounds. Because of the high amounts of organic matter and macronutrients (especially potassium), OMW could be considered as a useful, low cost amendment and fertilizer (Proietti *et al.* 1995; Bonari *et al.* 1993) [3,24].

In the present paper, we investigated the effects of OMW spreading on the chemical characteristics of soil cultivated with vine, at different depths (10–30 cm and 30–60 cm*)*. The possibility of reducing the use of chemical fertilizers was also considered.

2. Material and methods

2.1. Experimental plots

The experiment was carried out in a vineyard (cv. *Italia*), 4 years old, installed in the Mellahi field, with a surface of 8 hectares and located in the Meknès area. Nine contiguous and homogeneous land plots, of approximately 24 m^2 each, were subjected to spreading with different amounts of OMW, as the following: three land plots with 10 L/m^2 (T_1 treatment), three with 20 L/m^2 (T_2 treatment) and the remaining three land plots were used as witness (T_0). Cultural practices were identical in all land plots, which were separated from each other by a line of an untreated vineyard.

2.2. Sampling

Samples of OMW were taken during 2 consecutive crop years (2005/2006 and 2006/2007), from a production facility equipped with a three phase system, and located in the Meknès area (Zouina Field, Agouray). The samples were taken from basin of OMW storage and were transported in cans of 5 L, and then stored at 4°C.

Soil samples were taken from a field of vineyard in the Meknès area. Three samples were taken along the diagonal of the elementary land plots, from the upper 10 cm of soil depth (sample 1) and in the 30–60 cm of depth (sample 2). Thus, total number of the samples is 6 per elementary land plot. The same sampling procedure was carried out for all the nine elementary land plots.

To assure homogeneity, the samples from the same land plot and depth were mixed together. A subsample of 1 kg was then taken and stored at 4°C, awaiting for analyzes.

Soil sampling was carried out at three periods, as follows: first period, before the spreading of the OMW (pilot); second period, 10 days after spreading; and third period, 60 days after spreading. The granulometric characteristics of the soil are shown in Table 1.

2.3. OMW characterization

The *pH* was measured using a glass electrode and was calibrated beforehand (*pH* 4 and 7). Electric conductivity (*EC*) was measured by a standard conductimeter of the Tacussel type. The suspended matter was determined by filtration on filters of glass. The drying of the filter was done at 105°C during 4 h, followed by a weighing (AFNOR NF T90–105). Totals polyphenols' content

Table 1
Granulometric characteristics of the soil.

Depth of the soil (cm)	Clays (%)	Silts		Sands	
		Fine silts %	Coarse silts %	Fine sands %	Coarse sands %
10–30	21.92	22.20	10.25	16.83	28.80
30–60	34.67	25.52	6.65	11.23	21.90

was determined by colorimetric method using Folin–Ciocaleu's reagent (Macheix *et al*. (1990) [25]. Oil content was determined by extracting the dry material with 40–60°C petroleum ether using a soxhlet apparatus (Di-Giovacchinon, 1986) [26]. OMW samples were diluted to the 10^{-3} (AFNOR T 90–101). Total nitrogen (NTK) was determined by Kjeldahl method, while available forms of phosphorus (P_2O_5) and exchangeable potassium (K_2O) by atomic absorption spectrometry. The calcium cations (Ca^{2+}) were proportioned by EDTA in alkaline medium (pH: 12), in the presence of murexide, as an indicator.

2.4. Soil analyses

2.4.1. Physicochemical determinations

Particle size distribution of soil was determined by pipette analysis with dispersion by sodium hexametaphosphate and classified according to soil survey staff. The pH was measured using a glass electrode at water–soil ratio 2.5:1 (*V/W)*. The water content was determined after drying at 105°C. Electric conductivity of the soil aqueous extract (1:25) was measured with a traditional conductivimeter. The organic carbon (C) was determined following the Walkley–Black method and organic matter was calculated by multiplying total carbon by 1.724 (Conseil des productions végétales du Québec, Agdex 533, Méthode MA-1, june 1988) [27]. Total nitrogen (N) was determined by Kjeldahl method, while available forms of phosphorus (P_2O_5) and exchangeable potassium (K_2O) by atomic absorption spectrometry. These analyses were performed according to standard methods of the "Ministero delle Politiche Agricole e Forestali, 2000 b" [28]. The total phenols content was determined by using Folin Ciocalteu's reagent on the same aqueous solution obtained for the pH determination (Macheix *et al*. 1990) [25]. The calcium carbonates were determined by the following method: Chlorhydric acid (HCl) was added to a sample of the soil, to break up the carbonates. The volume of carbonic gas (CO_2) emitted was measured using a "Bernard Calcimeter" and was compared to the volume of carbonic gas produced by pure calcium carbonates.

3. Results and discussion

The infiltration of OMW through soil layers induces changes in its physicochemical characteristics. The composition of OMW utilized for the spreading is reported in Table 2.

3.1. Soil pH

The application of OMW on an alkaline soil with amounts of 10 L/m^2 and 20 L/m^2 induces only a slight reduction in the soil pH.

After 10 days of OMW spreading, the pH was slightly lower in the upper soil layers (10–30 cm), due to the high acidity of OMW (Table 2). However, with high amounts of OMW (20 L/m^2) at the depth of 30–60 cm, the soil pH was slightly higher than the control. This change was due to production of ammonia following bacterial breakdown of organic matter present in OMW and to the high amount of potassium entering into the clay exchange complex of the soil, producing alkaline hydrolysis reactions. These results are confirmed by other authors (Proietti *et al*. 1995; Marsilio *et al*. 1990; Di Giovacchino *et al*. 2005). [3], [11], [10]. In a similar way, Bonari *et al*. (1993) [24] also showed that there exists a strong correlation between the reduction in soil pH and the amount of OMW applied. After 2 months of spreading, the soil *pH* tends to be stamped.

3.2. Electric conductivity (EC)

An increase in EC was observed in OMW treated soils, after 10 days of spreading. After 2 months, this parameter

Table 2
Physicochemical characterization of OMW obtained by centrifugation, in the Meknès area.

Parameters	Countryside 2005/2006	Countryside 2006/2007
pH	4.87	4.98
E.C (mS/cm/ 20°C)	4.80	5.22
Suspended matter (g/L)	1.98	2.11
COD (g O_2/L)	89.50	80.00
Phosphorus (P_2O_5) (mg/L)	240.00	319.00
Potassium (K_2O) (mg/L)	2300.00	3100.00
Calcium (Ca^{2+}) (g/L)	0.98	1.02
Total Nitrogen (NTK) Kjeldahl (mg/L)	280.00	330.00
Residual Oil (g/L)	1.43	1.50
Total polyphenols (caffeic acid, g/L)	7.50	6.30

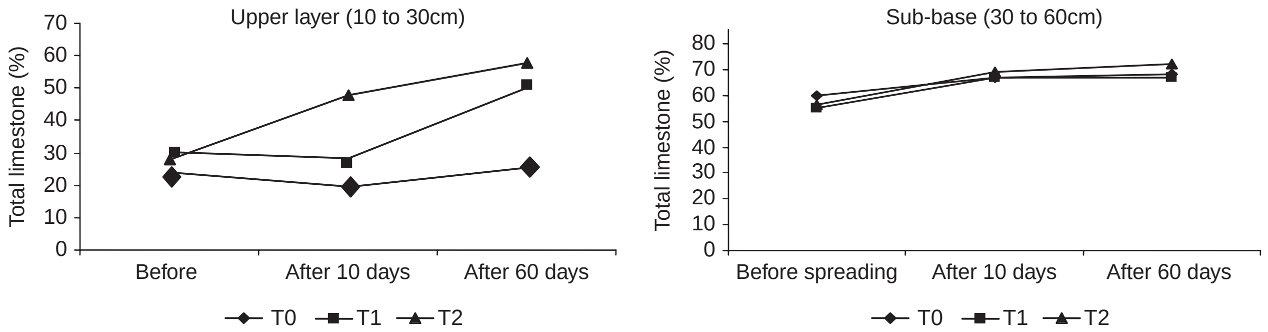

Fig. 1. Evolution of total limestone in the various soil layers, before and after spreading of OMW. .

returned to its initial values, except in the upper soil layer treated with 10 L/m² of OMW, where it appears to be irreversible. This effect was observed by other authors (Paredes *et al.* 1987; Tomati and *al.* 1992; Ben Rouina 1994; Di Giovacchino *et al.* 2005) [29–31], [10]. According to Levi-Minzi *et al*. (1992) [21], the application of OMW, up to 32 L/m² , led to the same results; which thus encourages the spreading of OMW on arable lands.

3.4. Total limestone (C_aCO_3)

The soil total content of limestone, in the upper layer, showed a considerable increase after spreading (Fig. 1). After 10 days of spreading, the average contents were about 27.5% and 48% respectively for the two treatments T_1 and T_2 against 20% for the witness (T_0). After 60 days, these values reached 50 and 56% respectively for T_1 and T_2, compared to the witness. OMW concentration in calcium, as well as the strong content of the soil limestone, favoured the precipitation of limestone in the form of calcium carbonate (Table 2). This might explain the increase observed in the percentage of soil total limestone, after spreading**.** At upper soil levels, a such increase was not observed, which might result from a retention of calcium ions (Ca^{2+}) by the carbonates ions (CO_3^{2-}) of the upper soil layer. These results were confirmed by other authors (Abichou, 2003; Ben Rouina *et al*. 2008) [32–33].

3.5. Organic matter

Percentage of organic matter in the soil upper layer also underwent a considerable increase after 10 days of spreading; but it tends to decline after 60 days. After 2 months of spreading, no significant difference was observed between treated soil samples and the witness. A such result can be explained by the fact that organic matter brought by OMW (Table 2) consisted mainly of easily biodegradable substances, which seldom come to be added to the humic reserve of the soil (Baize 2000; El Hassani *et al*. 2005) [34–35]. Similar results were recorded at the soil upper layer, where organic matter was retained due to the high moisture-holding capacity of the clay soil.

3.6. N.P.K

The contents of fertilizing elements (N.P.K) of the treated soil increased in a more significant way, especially for total phosphorus. At the soil upper layer, and after 10 days of spreading, the potassium contents clearly increased by 91 and 170 ppm respectively for T_1 and T_2 compared to T_0. After 2 months, the recorded average values were about 140 and 96 ppm for treatments T_1 and T_2 respectively, compared to the witness (Fig. 2). The reduction in potassium content following the spreading with 20 L/m² can be explained by the importance of the

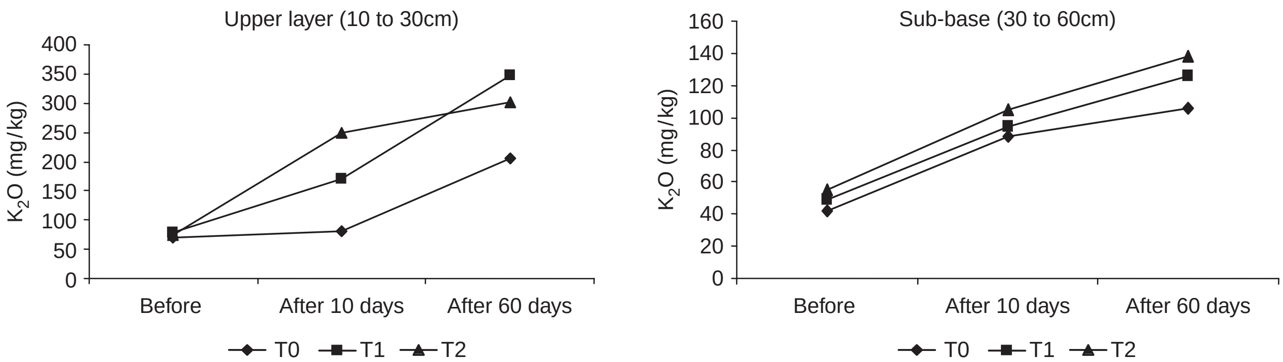

Fig. 2. Potassium evolution in the various soil layers, before and after spreading of OMW.

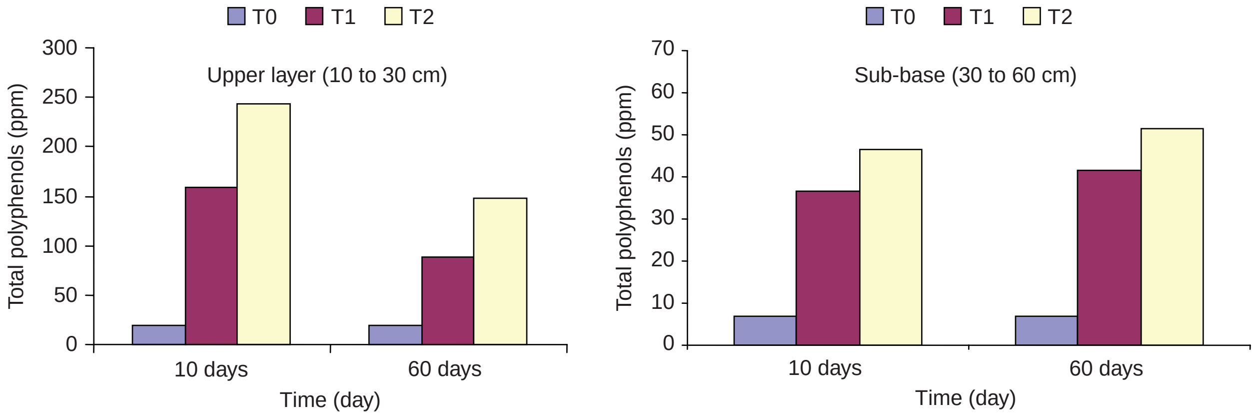

Fig. 3. Evolution of total phenols in the various soil layers, after spreading of OMW.

soil microbial activity and by the degradation of organic matter (Levi-Minzi *et al.* 1992) [21]. At the lower soil level (30–60 cm), the potassium content showed only a slight increase following spreading with 20 L/m² compared to the witness (40 and 100 ppm respectively after 10 days and 2 months of spreading). Similarly, the increases observed with 10 L/m² were of 5 and 20 ppm respectively for the two treatments T_1 and T_2. According to these results, it arises that the effect of OMW spreading on the exchangeable potassium content of the soil occurred primarily in the upper layer. At the lower soil level, the weak potassium increase can be explained by its adsorption by the dry humic complex and its integration inside the organic compounds. Phosphorus contents, initially comparable to those in the soil upper layer, were multiplied after 10 days by 1.8 and 2.1, respectively for the two treatments T_1 and T_2. High correlation was observed between phosphorus accumulation and the OMW amounts used for spreading ($R^2 = 0.93$ for T_2 and $R^2 = 0.89$ for T_1). At the end of 2 months, the easily assimilated phosphorus contents of the soil were multiplied by 1.7 and 1.5 respectively for T_1 and T_2 compared to T_0. Such increase was not observed in lower soil layers.

In the upper soil layer (10–30 cm), OMW spreading induced a big raise in total nitrogen. After 10 days of spreading, the contents are much higher than those of the witness. Nitrogen concentrations stabilized in lower soil layers since no significant difference was observed between the witness and treated land plots. This tendency can be explained since nitrogen present in the OMW is entirely in organic form, which is degradable by the soil biological activity (Di Giovacchino *et al.* 2001) [18].

The richness of OMW in N.P.K (Table 2) explains its valuation as fertilizer in cultivated soils (Morisot and Tournier 1986; Tomati *et al.* 1996; Paredees *et al.* 1987; Levi-Menzi *et al.* 1992) [36–37], [29], [21]. OMW thus represents a source of nutritive elements which could replace conventional fertilizers. Indeed, 1 m³ of OMW from a three phase plant brings, on average, 760 g of N.T.K and 633 g of phosphorus (Morisot and Tournier 1986) [36]. The content of residual oil in soil, brought by OMW, remained weak or even nil.

3.7. Total phenols

The concentration of reducing substances (total phenols) increased just after 10 days of OMW spreading. 2 months after OMW spreading, the total content of phenols was reduced by about 80 and 100 ppm compared to the values recorded after 10 days (*Fig*. 3). This reduction is probably due to their incorporation into the humic fraction of the soil organic matter and their breakdown, caused by specific bacteria and yeasts (Borja *et al.* 1994; Martirani *et al* 1996) [38–39]. This biological breakdown of phenols immobilized at the upper soil layer was supported by the neutral pH and ventilation. The major part of phenols was retained at the upper soil layer and a slight migration took place towards the lower layers.

4. Conclusion

The pH, after 10 days of OMW spreading, was slightly lower in the upper soil layers (10–30 cm), due to the high acidity of OMW. However, with increased amounts of OMW (20 L/m²) at the soil depth of 30–60 cm, pH was slightly higher than the control. This change was due to production of ammonia following bacterial breakdown of organic matter present in the OMW and to the high amount of potassium entering into the clay exchange complex of the soil, producing alkaline hydrolysis reactions. After 10 days of spreading, an increase in electric conductivity was observed. This parameter returned to initial values, after 2 months of spreading, except in the upper soil layer treated with 10 L/m^2 of OMW, where it appears to be irreversible. After 10 days of spreading, the percentage of organic matter in the treated soil has undergone an increasing evolution at the upper soil layers. But it tends to be stamped after 60 days because of a biological breakdown in products having humic characteristics. On the other hand, at the lower soil layers, no effect of OMW spreading was recorded. This could be explained by retention of organic matter at the upper soil layer, thanks to the high moisture-holding capacity of the clay soil. The concentration of calcium ions in OMW, as well as the strong content of the soil limestone, favoured the precipitation of limestone in the form of calcium carbonate. The increase in the content of K and P (expressed as exchangeable potassium and available phosphorus) had a beneficial effect on soil fertility.

Two months after OMW spreading, the total content of phenols was reduced, probably due to their incorporation into humic fraction of the soil organic matter and to their breakdown caused by specific bacteria and yeasts. The results of this experiment confirm the economic and environmental validity of the OMW spreading since it did not induce toxic effects on cultivated soils, and helped to reduce the amount of chemical fertilizers.

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Acronyms

OMW:	olive mill wastewater
T:	thunder
ppm:	parts per million (mg/kg)
P_2O_5:	available form of Phosphorus
K_2O:	available form of Potassium
Ca^{2+}:	calcium ions
pH:	hydrogen potential
EC:	electric conductivity
EDTA:	ethylene diamine tetra acetic acid
COD:	chemical oxygen demand
NTK:	total nitrogen
$CaCO_3$:	calcium carbonate (limestone)
cv:	cultivar
T_0:	witness
T_1:	treatment with an amount of 10 L/m^2
T_2:	treatment with an amount of 20 L/m^2
R^2:	coefficient of determination
v/w:	ratio (volume/weight)

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