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Long term effects of olive mill wastewaters application on soil properties and phenolic compounds migration under arid climate

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ABSTRACT

The principal objective of the present study has been to investigate the long term application effects of olive mill wastewaters (OMWW) on the main properties of a Mediterranean soil under arid climate. In addition the migration dynamic and evolution of phenolic compounds in the different soil layers over time and according to the OMWW amount added have been studied.

Results have showed that the irrigation of sandy soils by different doses of OMWW has influenced the soil physicochemical and microbiological characteristics. However, a slight change in the granulometric composition of soil treated with OMWW has been distinguished. The study of the phenolic compounds dynamic has showed the migration of these compounds further than 120 cm soil depth. The comparison of the different spectra obtained by the high pressure liquid chromatography (HPLC) technique has explained that the high molecular weight polyphenols which have been retained in the upper soil layers. However, the low molecular weight phenolic compounds have been detected in depth. All the results obtained have proved that the dose of 50 $m^3\,ha^{-1}$ has been the most suitable for the soil studied.

1. Introduction

The world's olive-growing heritage covers 9.5 million hectares with more than 900 million olive trees, of which almost 95% are in the Mediterranean basin (IOC, 2015). Indeed, olive growing plays a major role in the economy of Mediterranean countries, especially Spain, Italy, Tunisia and Greece (Magdich et al., 2016).

Olive growing in Tunisia is a strategic socio-economic sector. Currently, Tunisia's olive-growing potential is estimated at nearly 90 million trees, occupying an area of 1.8 million hectares or 79% of the total arboreal area (GDAP, 2015). Indeed, Tunisia contributes by 6% of the world production, nearly 60% of the African production and 9% of the world exports (Karray et al., 2010; FAO, 2015; GDAP, 2015).

Olive mill wastewater (OMWW) is the liquid by-product generated during olive oil production. The annual production of OMWW in Mediterranean countries reached 30 million

cubic meters and 700 000 cubic meters in Tunisia alone (Mekki et al., 2013; Ben Rouina et al., 2014). Thus, the management of OMWW poses serious environmental and economic problems for all olive oil producing countries including Tunisia (Mechri et al., 2011; Mekki et al., 2013; Magdich et al., 2016).

OMWW are characterized by an intense purple-brown to black color and an odor of olive oil (Saadi et al., 2007; Daâssi et al., 2014). These effluents have an acidic pH with values between 3.5 and 5.5 and elevated water content with percentage from 83 to 95% (Nasini et al., 2013; Magdich et al., 2016). OMWW generally have a high salinity due to the important addition of salt for the conservation of olives (Hachicha et al., 2009). The mineral fraction consists mainly of potassium, which has led several researchers to test their fertilizing power (Piotrowska et al., 2011; Killi and Kavdir, 2013). The OMWW organic matter comprises an insoluble fraction consisting essentially of pulp of olives, suspended matter and colloidal, and a soluble fraction in the aqueous phase which contains sugars, lipids, organic acids, pectins, phenolic compounds, vitamins and traces of pesticides (El Hadrami et al., 2004; Komilis et al., 2005; Kapellakis et al., 2008).

OMWW phenolic compounds are diverse and their structure is very variable (Allouche et al., 2004). These phenolic compounds are divided into low-molecular weight (caffeic acid, tyrosol, hydroxytyrosol, p-cumaric acid, ferulic acid, syringic acid, protocatechuic acid) and high molecular weight compounds (tannins, anthocianins) (Allouche et al., 2004; Davies et al., 2004).

In Tunisia, the climatic aridity, the poverty of soils in organic matter and based on several previous works (Ben Rouina et al., 2014; Mekki et al., 2007, 2013) have led the authorities to opt for the use of OMWW

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in the agricultural sector as soil organic fertilizers (Decree 1306 of February 26, 2013). This type of irrigation acts on the rhizosphere and more specifically on the microorganisms associated with this part (Mechri et al., 2009; Piotrowska et al., 2011; Mekki et al., 2013).

Soils irrigated by OMWW provide a favorable environment for the development of soil microflora, which means recycling organic matter and enriching mineral elements that increase soil fertility (Sierra et al., 2007; Mekki et al., 2009; El Hassani et al., 2010). Thus, an improvement of the respiratory potential and a reduction of the evapotranspiration have been observed following the use of OMWW (Mekki et al., 2012; Abichou et al., 2014). However, there is still a lack of knowledge on the impact of the use of OMWW on the evolution of phenolic compounds in the soil and this depending on the amount applied and the time of application (Mekki et al., 2007).

The objectives of this study are based on previous works, the results of which led to the agronomic valorization of the OMWW in Tunisian soils amendment under arid Mediterranean climate (Decree 1306 of February 26, 2013). More specifically, we will focus on the study of the long term application effects of OMWW on the main properties of a soil from the region of Sfax, South of Tunisia. Moreover the migration dynamic and evolution of phenolic compounds in different soil layers over time and according to the amount of OMWW applied to the soil have been investigated.

2. Materials and methods

2.1. Olive mill wastewaters origin and sampling

The olive mill wastewaters used for this work comes from a continuous three-phase extraction system (olive oil extraction factory located in the region of Gremda in Sfax, Tunisia). OMWW have been brought back and stored in a basin at the experimental station "Taous" of the Olive Tree Institute (Sfax, Tunisia) and spread on the soil. Homogeneous samples of OMWW have been brought back to the laboratory for analyses.

2.2. Soils description and sampling

In order to study the long-term effects of irrigation of OMWW, three plots (P1, P2 and P3) with an area of 1 ha each, located at the experimental station "Taous" of the Olive Tree Institute (North latitude 34° 3′, East longitude 10° 20′, the average annual precipitation and temperature over 15 years have been 210 mm and 25 °C respectively) and which have been spread by raw OMWW at December each year, since 2004. P1 has been considered as a control plot (without irrigation), P2 has been irrigated with 50 $m^3 ha^{-1}$ and P3 has been irrigated with 200 $m^3 ha^{-1}$. The last application has been done in December 2016. After 2 months of the last OMWW application (February 2017), soils sampling have been done at different depths; 0–30, 30–60, 60–90, 90–120 and 120–150 cm (the soil sampling has been carried out at the rate of 5 points per plot, with three repetitions per soil depth level (ie overall 15 samples per soil depth level which corresponded to 75 samples per plot). Samples from different layers and soils have been brought to the laboratory for analyses.

2.3. Olive mill wastewaters sampling and characterization

For all OMWW applied to soils since 2004, 5 homogeneous samples have been taken from different points and depths of the OMWW storage basin. All samples have been brought to the laboratory and stored at 4 °C for analyses.

The main physicochemical characteristics of all OMWW samples have been determined. The pH and electrical conductivity (EC) have been resolute according to Sierra et al. (2007). OMWW water content and the dry matter content have been dogged by drying at 105 °C for 24 h. Organic matter and mineral matter contents have been calculated after heating the samples at 550 °C in a laboratory furnace for 4 h. The organic matter has been evaluated by difference between dry matter (after drying at 105 °C) and mineral matter (after calcination at 550 °C). For the chemical oxygen demand (COD), the Knechtel method (1978) has been used. Biochemical oxygen demand (BOD_5) has been measured by the manometric method with a respirometer BSB-Controller, Model 620 T.WTW. The OMWW phenolic compounds contents have been quantified according the Folin-Ciocalteau method (Box, 1983). Total nitrogen content has been determined according to the Kjeldahl method (Knechtel, 1978). OMWW Phosphorus, iron, magnesium, potassium, sodium and chloride have been analyzed by atomic absorption using Hitachi U-2000 spectrometer.

2.4. Soils granulometric analyses

The granulometric composition of different soils samples has been determined by the international Robinson pipette method, based on Stokes' law that "the larger the particle, the faster it falls into the water" (Stanislaw and Wojciech, 2013). The soil sample has been placed in a specific solution (H_2O_2) which destroys the aggregates. The particles have been then separated by sieving for the higher fractions and by gravity sedimentation for the finer ones. A known volume of the suspension has been taken and stored in a crucible. The solid residue has been weighed after evaporation. Fractions obtained by sieving make it possible to determine the proportions of sand. The fractions obtained by pipetting give the proportions of fine silt and clay.

2.5. Soils physicochemical analyses

The pH and electrical conductivity (EC) of each soil sample have been determined according to Paredes et al. (1987) standard method. Soils water contents, dry matters (DM), organics matters (OM) and mineral matter (MM) have been evaluated according to Sierra et al. (2007) standard method. Soil water retention capacity (SWRC) has been measured according to a standard method (SALTERS and WILLIAMS, 1965). Soils total nitrogen contents have been assessed by Kjeldahl method (Kandeler et al., 1995). Available phosphorus (P) and potassium (K) have been analyzed following the method cited by Magdich et al. (2013).

2.6. Soils respirometric activities analyses

For each soil sample (soil control (Sc), soil irrigated with OMWW at dose of 50 $m^3 ha^{-1}$ (S_{50}) and soil irrigated with OMWW at dose of 200 $m^3 ha^{-1}$ (S_{200})) and for all depths, microbiological activities have been assessed by respirometric activities measurement according to standard method (Ohlinger et al., 1995).

2.7. Soil phenolic compounds analyses

For all soils samples, water soluble phenolic compounds have been extracted and determined according to a standard method (Allouche et al., 2004). In fact, for each soil sample, a 1/25 (w/v) soil/aqueous mixture has been shaken for 10 h in a mechanical shaker. The supernatants have been extracted 3 times with ethyl acetate. The collected organic fractions have been dried and evaporated under vacuum. The residues have been extracted twice with dichloromethane in order to remove the non-phenolic fraction (lipids, aliphatic and sugars). The liquid phases have been discarded while the washed residues were analyzed by the Size exclusion-HPLC technique.

2.8. Statistical analyses

Statistical analyses of all the parameters studied have been carried out using SPSS software (Statistical Package for the Social Sciences, version 20). The experiments have been performed in triplicate. They