



Impact of spreading olive mill waste water on agricultural soils for leaching of metal micronutrients and cations



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HIGHLIGHTS

- OMWW is frequently spread on agricultural land for disposal.
- OMWW effect on leaching of indigenous soil metals was tested in four soils.
- OMWW contributed metals to the soil solution.
- OMWW mobilized indigenous soil metals as a function of soil characteristics.
- Metal leaching was attributed to cation exchange and organic-metal complex formation.

ARTICLE INFO

Article history:

Received 8 January 2017

Received in revised form

8 March 2017

Accepted 22 March 2017

Available online 25 March 2017

Handling Editor: T Cutright

Keywords:

Olive mill wastewater

OMWW

Metals

Soil spreading

Cation exchange

Complex formation

ABSTRACT

Olive mill waste water (OMWW) is an acidic (pH 4–5), saline (EC ~ 5–10 mS cm⁻¹), blackish-red aqueous byproduct of the three phase olive oil production process, with a high chemical oxygen demand (COD) of up to 220,000 mg L⁻¹. OMWW is conventionally disposed of by uncontrolled dumping into the environment or by semi-controlled spreading on agricultural soils. It was hypothesized that spreading such liquids on agricultural soils could result in the release and mobilization of indigenous soil metals. The effect of OMWW spreading on leaching of metal cations (Na, K, Mg, Mn, Fe, Cu, Zn) was tested in four non-contaminated agricultural soils having different textures (sand, clay loam, clay, and loam) and chemical properties. While the OMWW contributed metals to the soil solution, it also mobilized indigenous soil metals as a function of soil clay content, cation exchange capacity (CEC), and soil pH-buffer capacity. Leaching of soil-originated metals from the sandy soil was substantially greater than from the loam and clay soils, while the clay loam was enriched with metals derived from the OMWW. These trends were attributed to cation exchange and organic-metal complex formation. The organic matter fraction of OMWW forms complexes with metal cations; these complexes may be mobile or precipitate, depending on the soil chemical and physical environment.

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1. Introduction

Soil serves as a medium for organic waste disposal, both solid and liquid (Marshall et al., 1996). Uncontrolled spreading of liquid organic wastes, in particular, may pose pollution risks including contamination of the soil and soil profile, surface waters, and groundwater. One organic liquid waste that is commonly land spread is olive mill waste water (OMWW). OMWW is a blackish-red

aqueous byproduct (90–95% water) of the three-phase olive oil production process. OMWW chemical characteristics vary depending on the particular olive cultivar and olive oil production process, but generally have similar characteristics (Aviani et al., 2012): acidic pH (4–5), high salt content (EC ~ 5–10 mS cm⁻¹), extremely high biological (BOD) and chemical oxygen demand (COD) (up to 100,000 and 220,000 mg L⁻¹, respectively), and high concentrations of organic compounds such as polyphenols (Azbar et al., 2004; Aviani et al., 2009), carbohydrates, polyalcohols, tannins and pectins (Poerschmann et al., 2013a, 2013b). OMWW also contains high concentrations of potassium, magnesium, sodium,

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calcium and phosphate (Arienzo and Capasso, 2000). Therefore, OMWW cannot be discharged into domestic waste water treatment plants (Rozzi and Malpei, 1996). The most common disposal practice for OMWW is poorly-controlled land spreading to cultivated or uncultivated fields (Kapellakis et al., 2008), or to OMWW lagoons and constructed wetlands (Kontos et al., 2015). Other biological and physicochemical treatments are also employed (Kontos et al., 2015; Pulido, 2016; Ochando-Pulido et al., 2017). In Israel, land spreading is by far the most common disposal practice as other treatments have been found to be prohibitively expensive (Laor, 2009). OMWW spreading on soil is known to cause phytotoxicity (Mekki et al., 2006; Saadi et al., 2007), soil salinization (Paredes et al., 1987; M. Hameed and Aqeel, 2007), and soil water repellency (Graber et al., 2009). Land disposal of OMWW induces a significant increase in water-soluble and exchangeable potassium (Gallardo-Lara et al., 2000).

Generally, reducing soil pH while elevating dissolved organic matter (DOM) content can result in mobilization of heavy metals (Förstner and Wittmann, 2012). Specifically, the acidic nature of OMWW was found to cause local reversible decreases in soil pH (Cabrera et al., 1996; Piotrowska et al., 2011), which resulted in release of heavy metals from soils contaminated with metals-rich mine-tailings (Bejarano and Madrid, 1992, 1996b). Metals in mining-contaminated soils were also released due to formation of heavy metal complexes with the water soluble organic compounds in the OMWW (Bejarano and Madrid, 1996a; Díaz-Barrientos et al., 1997), for example, Cu and Zn (Madrid and Díaz-Barrientos, 1998). Release of other elements such as Fe and Mn was promoted mainly by redox reactions (Colarieti et al., 2006; Greco et al., 2006; Piotrowska et al., 2011). In contrast, soil amendment with solid composted olive mill waste significantly reduced the release of metals (Madrid and Díaz-Barrientos, 1998). These studies all addressed release of metals from heavily contaminated soils. In most cases, however, OMWW is not spread on contaminated lands but on agricultural lands. While the impact of OMWW spreading on physical, biological, and chemical attributes of agricultural soils has been widely explored (Piotrowska et al., 2006, 2011; Di Bene et al., 2013), the question of release of soil-borne metal cations has not been addressed.

The behavior of soil metals following OMWW application on non-contaminated agricultural lands may differ substantially from that in contaminated soils, as the assemblage, speciation and associations of the metals will be quite different. The focus of the current study was to examine the effect of OMWW spreading on non-contaminated agricultural soils on leaching of indigenous soil metal cations, by mimicking the conventional agricultural practice of spreading OMWW on the soil surface. Such spreading is followed after some time by rainfall or irrigation. Four main questions were addressed: (i) does saline OMWW spreading change the metal cation content of the soil solution; (ii) does it cause mobilization of indigenous soil metals; (iii) in case of mobilization, what are the mechanisms; and (iv) how do soil characteristics influence metal cations leaching.

2. Materials and methods

2.1. Soils

Four Israeli agricultural soils having a wide range of chemical and physical properties and soil textures were selected (Aharonov-Nadborny et al., 2016): red sand (sand soil); brown loam (loam soil); grumusol (clay soil); and loess (clay loam soil). (Table S1). Also presented in Table S1 are the cation exchange capacity (CEC), exchangeable sodium percentage (ESP), and exchangeable potassium percentage (EPP) (Rhoades, 1982), as well as available major

cations (Ca, Mg, Na and K) of the saturated paste soil solution (Rowell, 1994).

2.2. Amendment solutions

Olive mill wastewater (OMWW) was provided by the Bnei-Darom, Israel, olive oil production facility (Aharonov-Nadborny et al., 2016). In addition to OMWW (filtered via 20–25 µm cellulose paper filter (Whatman 41)), a salt solution having Ca, Mg, Na, K and phosphate contents similar to that of the OMWW but without organic matter was used as a control treatment (Aharonov-Nadborny et al., 2016), as was de-ionized water (DW). Solution characteristics are given in Table S2.

2.3. Columns setup and experimental procedure

Metal leaching was tested in columns following the method previously described by Aharonov-Nadborny et al. (2016). In brief, there were four sets of 60 ml polyethylene disposable syringe barrels (2.8 cm i. d. x 11.5 cm length) packed with methanol-cleaned glass wool (2 cm at the bottom), 20 g of pretreated quartz sand (1.5–2 mm particles), 40–45 g of test soil, cellulose paper filter (Whatman 41) disc, and another 10 g portion of the pretreated sand on top (total soil + sand height was 7 cm). Pretreatment of the quartz sand included heating to 450 °C for 24 h, serial washing with a reducing solution, acid solution and DW, and then oven drying. The columns (six replicates per treatment) were wetted with 1 pore volume of each amendment solution: OMWW, salt solution, and DW, after which they were left overnight at room temperature. The columns were washed by passing five portions of 3.0–3.5 pore volumes per portion (approx. 50 mL) of 0.2 M calcium chloride solution, aided by a laboratory vacuum pump. The total time for leaching of each portion was 15 min. All leachates were collected, filtered (<0.45 µm), and analyzed for concentrations of metal ions, dissolved organic carbon (DOC), PO₄, chloride, NH₄ and NO₃.

The 0.2 M CaCl₂ solution was employed as the eluent in order to reduce clay swelling in the soil columns. In part, exchange of cations between the soils and the eluent contributed to metals' leaching. However, the use of the eluent was consistent for all the soils and the various treatments, and it is anticipated that its effect on metals' leaching between the treatments for a given soil will be similar.

2.4. Analytical methods

P-phosphate, N-ammonium, N-nitrate and chloride were measured colorimetrically using an autoanalyzer (Quickchem 8000, Lachat Instruments, Loveland, Colorado, USA). Dissolved organic carbon was determined using a TOC-VCPN instrument (Shimadzu Corp., Kyoto, Japan). Sodium and potassium concentrations were determined by flame photometer M410 (Sherwood Scientific Ltd, Cambridge, UK). Magnesium, manganese, iron, copper, and zinc were measured by atomic absorption spectroscopy (AAS; Analyst 800 atomic absorption spectrophotometer, Perkin Elmer, Waltham, Massachusetts, USA). Metal cations speciation was calculated using Visual MINTEQ 3.0 software wherein DOC was modeled on the basis of the Stockholm humic acid model (Gustafsson, 2001). Computer calculations used temperature, pH, DOC, and measured dissolved cations and anions as data input.

2.5. Statistical analyses

Statistical analyses were done using SigmaPlot 12.0 software. The data was analyzed by one-way ANOVA. *Post-hoc* means

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