

Effects of amendment with treated and untreated olive oil mill wastewaters on soil properties, soil humic substances and wheat yield

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Received 25 January 2006; received in revised form 23 October 2006; accepted 7 November 2006

Available online 19 December 2006

Abstract

Catalytic digestion with MnO₂ was shown to be a suitable treatment for promoting organic matter humification in olive oil mill wastewater in order to increase its potential as a soil amendment and reduce environmental risk. In this study, the effects of addition of either lagooned wastewater (LW) or catalytically digested wastewater (CW) at two rates on soil and soil humic acid (HA) properties and durum wheat yield were investigated in a field experiment. Amendment with LW or CW increased soil electrical conductivity and contents of total organic C, total extractable C, humified and non-humified C forms, and available P and K. In comparison to LW-amended soils, CW-amended soils featured larger contents of total extractable C and humified C. The compositional, structural and functional characteristics of soil HA were modified by the partial incorporation of HA-like components typical of applied LW and CW. In particular, amended soil HAs featured an increase of C, H and phenolic OH group contents, C/N ratio and aliphaticity, whereas C/H ratio, N, O and COOH group contents, aromatic polycondensation and humification degree decreased. These modifications were generally more evident in HAs from LW-amended soils than in HAs from CW-amended soils. Addition of LW, and especially CW, affected positively the wheat grain yield by increasing the number of kernels per unit area, spike density, and kernel weight. In conclusion, the catalytic treatment of olive oil mill wastewater with MnO₂ is able to produce an efficient soil amendment.

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Keywords: Olive oil mill wastewater; Soil amendment; Soil properties; Soil humic acids; Wheat yield

1. Introduction

Sustainable agricultural practices based on the periodical input of appropriate organic amendments to soil are encouraged in Mediterranean agro-ecosystems that are naturally poor in soil organic matter and greatly exposed to severe risks of erosion and desertification (Senesi, 1989; Lal, 2001; Plaza et al., 2002; Brunetti et al., 2005). In particular, the high mineral and organic content of olive oil mill wastewater (OMW), which is an abundant by-product of the olive oil industry in Mediterranean countries, renders this effluent potentially suitable for recycling as a soil amendment (Paredes et al., 1999; Rinaldi et al., 2003). The OMW consists of the water added during the oil extraction

process, the aqueous fraction and portions of pulp tissues originating from olives, and a stable emulsion of oil residues (Paredes et al., 2002; García-Gómez et al., 2003).

The OMW is commonly stored in open-air lagoons before its application to soil without any further treatment (Sierra et al., 2001; Komilis et al., 2005). However, the application of insufficiently stable organic matter may induce a number of negative effects on soil properties and plant growth, such as the increase of mineralization rate of native soil organic C, induction of anaerobic conditions, release of phytotoxic substances, and microbial immobilization of plant nutrients (Senesi, 1989; Cereti et al., 2004; Komilis et al., 2005). Thus, prior to application to soil the fresh OMW should be subjected to appropriate treatments in order to stabilize its organic matter through humification, improve its efficiency as an organic amendment, and reduce its environmental risk.

Manganese oxides are common soil minerals that can act as abiotic catalysts in several reactions responsible for organic

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matter humification under natural conditions (Shindo and Huang, 1982, 1984; Huang, 2000; Jokic et al., 2001, 2004a,b). These include condensation between sugars and amino acids and oxidative polymerization of phenols leading to the formation of N-containing polycondensates and polymers. Further, results of previous studies (Senesi et al., 1999; Brunetti et al., in press) have shown that catalytic digestion with Mn(IV) oxide under ambient conditions is an efficient treatment to induce organic matter humification in OMW.

The objectives of this work were to evaluate the effects of amendments with untreated (lagooned) OMW or catalytically digested OMW at two different rates on: (a) some relevant soil chemical properties; (b) the molecular, structural and functional characteristics of soil HAs; and (c) the yield of durum wheat crop.

2. Materials and methods

2.1. Olive oil mill wastewaters, soils and field experiment

The fresh OMW sample was collected from an olive oil mill located in the Bari province, Southern Italy, which employs a three-phase decanter centrifuge process for oil separation. Aliquots of 2000 L of fresh OMW were either left standing for 60 days in an open-air lagoon (LW) or catalytically treated for 60 days with 2 kg of 90–95% MnO₂ (RdH Laborchemikalien GmbH and Co., Germany) in a reactor with continuous air bubbling from the bottom and mechanical stirring (CW). Both treatments were conducted under ambient conditions.

The field experiment was conducted in Spinazzola (Bari province) on a sandy loam soil (sand, 672 g kg⁻¹; silt, 160 g kg⁻¹; clay, 168 g kg⁻¹) classified as a Typic Haploxerept (Soil Survey Staff, 2003) or Haplic Calcaric Regosol (FAO/ISRIC/ISSS, 1998). The site is characterized by a Mediterranean climate (UNESCO/FAO, 1963) with mild winters and hot and dry summers. The average annual rainfall is 490 mm, which occurs mostly in the autumn and winter, and the average annual temperature is 15 °C.

The experimental design includes nonirrigated plots (2 m × 2 m) arranged randomly, either unamended (S) or amended with LW and CW at rates of 300 and 600 m³ ha⁻¹ (LW300 and LW600, and CW300 and CW600, respectively). Each treatment consisted of four replicates. The LW or CW was applied in late March 2003, and immediately incorporated into the soil at a depth of 0–20 cm. Five surface soil subsamples (Ap horizon, 0–20 cm depth) were collected randomly from each plot in late July 2003 using a soil probe of 3-cm diameter. A soil sample was then obtained for each plot by mixing equal amounts of the five corresponding soil subsamples. In early November 2003, durum wheat (*Triticum turgidum* L.) was planted in rows 15-cm wide at a seeding rate of 250 kg ha⁻¹. During the growing season weeds were controlled by applying the herbicides clodinafop-propargyl (prop-2-ynyl(R)-2-[4-(5-chloro-3-fluoropyridin-2-yloxy)phenoxy]propionate) and tribenuron-methyl (2-[4-methoxy-6-methyl-1,3,5-triazin-2-yl(methyl)carbamoylsulfamoyl]benzoic acid) at a rate of 250 g ha⁻¹ and 15 g ha⁻¹, respectively. Soon after the wheat physiological

maturity was reached (mid-July 2004), the entire aboveground wheat biomass was collected from each plot.

2.2. Wastewater and soil analyses

The principal chemical properties of OMW samples were determined in triplicate by conventional methods (Clesceri et al., 1998). In particular: (a) dry matter was measured after heating the sample overnight at 105 °C; (b) ash content by burning the sample overnight at 550 °C; (c) pH with a glass electrode; (d) electrical conductivity (EC) by using a conventional conductivity cell; (e) total phenol content by the Folin-Ciocalteu method; (f) total organic C (TOC) by dichromate oxidation followed by titration with ferrous ammonium sulphate; (g) total N content by the Kjeldahl method; and (h) total P and K contents by inductively coupled plasma-atomic emission spectrometry (ICP) on nitric and perchloric acid digests of each sample.

The principal chemical properties of soils examined were determined by conventional methods (Sparks et al., 1996) on samples that were previously air-dried, crushed and passed through a 2-mm sieve. Briefly: (a) pH was determined at a ratio soil:water=1:2.5; (b) EC was measured on water extracts obtained at a ratio soil:water=1:5; (c) TOC and total N were measured by using the same methods as for OMWs (see above); (d) available P and K contents were determined, respectively, by the Olsen method and by ICP on 1 M BaCl₂ soil extracts using a ratio soil:extractant=1:10.

Total extractable C (TEC) and humified C (HA+FA) fractions were isolated from soils by using the procedure described in Ciavatta et al. (1988). Briefly, a mixture of 2 g of 0.5-mm ground soil sample was added with 100 mL of a 0.1 M Na₄P₂O₇ and 0.1 M NaOH solution, and shaken for 48 h at 65 °C. The supernatant solution was then separated from the residue by centrifugation and filtration. Twenty-five mL of the supernatant (TEC fraction) was acidified to pH ~ 1 with 50% H₂SO₄ to allow the precipitation of the HA fraction. The dissolved fulvic acid (FA) fraction was purified by passing the solution onto a column filled with polyvinylpyrrolidone, and then added to the HA fraction. The combined HA and FA fraction was transferred into a 50 mL flask and brought to volume with 0.5 M NaOH. The contents of TEC and (HA+FA)-C were determined by dichromate oxidation followed by titration with ferrous ammonium sulphate. The non-humified (NH)-C content was calculated by difference: NH-C = TEC - (HA+FA)-C. The degree of humification (DH) was calculated as DH% = 100 × (HA+FA)-C / TEC; the humification rate (HR) as HR% = 100 × (HA+FA)-C / TOC; and the humification index (HI) as HI = NH-C / (HA+FA)-C (Sequi et al., 1986; Ciavatta et al., 1988).

2.3. Isolation of humic acids

A conventional procedure (Schnitzer, 1982) was used to isolate the HA-like fractions from the OMW samples LW and CW and the HAs from each composite soil sample obtained by mixing equal weights of the four replicates of each treatment. Briefly, each air-dried, 2-mm sieved OMW and soil sample was

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