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Agricultural use of microfiltered olive mill wastewater: effects on maize production and soil properties

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Abstract

The disposal of olive mill wastewater (OMWW) is considered as one of the most serious environmental problems in the Mediterranean region. This study constitutes part of a holistic OMWW management approach aiming at the production of high added value products from OMWW with zero discharge. OMWW constitutes an organic material that could be recycled back to the soil after its treatment with microfiltration, and used as liquid fertilizer for plant production, thus leading to an environmentally friendly cultivation method with minor carbon footprint, since wastewater would (partially or fully) substitute mineral fertilizers, fresh water savings, and also economic benefits to the farmer.
Microfiltered OMWW (MF-OMWW) was applied to maize cultivation in a clay loam soil using two rates of 25 and 50 Mg ha^{-1}, with the addition of mineral fertilization of 200 kg N ha^{-1}. Furthermore, a treatment of only MF-OMWW applied at the rates of 50 Mg ha^{-1} and an only mineral fertilization treatment were used. The four treatments were replicated four times.
The results of the 1st year experiment showed that the different amounts of MF-OMWW used had no significant effect on soil properties. Maize yield, kernel moisture and fat content were not significantly influenced by the different treatments, whereas kernel protein, starch, fiber and ash content were significantly affected. Considering all quality and quantity parameters studied, the treatment with only mineral fertilizer N application gave similar results with the only MF-OMWW treatment, indicating the potential of mineral fertilizer full substitution by MF-OMWW, under the conditions of our study.

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Keywords: olive mill wastewater; microfiltration; liquid fertilizer; maize yield; kernel quality; clay loam soil

1. Introduction

Olive mill wastewater (OMWW) is a by-product of the olive oil extraction process that consists of vegetation water, and water used in the various stages of the oil extraction process [1]. OMWW constitutes a serious environmental problem in the Mediterranean area, due to its high polluting load, mainly referring to its high solids

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and organic compounds, high COD content, phytotoxic properties and resistance to biodegradation caused by its phenolic compounds [2, 3]. As a matter of fact, the pollution effect of 1 m^3 of OMWW is considered equivalent to 100 - 200 m^3 of domestic sewage.

As olive oil production in Greece is carried out mainly by small or small medium enterprises, the OMWW produced is usually applied untreated to nearby land, in order to avoid treatment costs. Research has shown that the application of untreated OMWW to agricultural soil may increase soil organic matter, available P and K [4], and total N content [5], but also it may increase soil electrical conductivity, salinity [5] and modify the equilibrium of useful soil microorganisms [6]. Moreover, it may result in germination problems due to phytotoxic effects of the phenolic compounds contained in the OMWW [7, 8]. Crop response in the OMWW applications is largely dependent on species sensitivity. Research work has shown that ryegrass and proteic pea yields were increased with untreated OMWW application, whereas clover yield was negatively affected [4]. Olive trees and also olive fruit yield and quality were not affected by OMWW application [9]. In another study, although maize growth was not affected, plant stress parameters were found increased following the untreated OMWW application [5].

On the other hand, the phenolic compounds contained in the OMWW are natural antioxidants, with commercial and economic interest. Membrane filtration of OMWW may lead to a significant decrease of its organic load and suspended solids content [2, 10], and also to separate polyphenols from the mass of waste [1, 11, 12]. OMWW treatment with microfiltration (MF) has shown that polyphenols can be effectively separated in the permeate [1]. Polyphenols may then be successfully removed with the use of suitable resins [13, 14]. As a result, the recovered polyphenols, may be utilized in the pharmaceutical, cosmetic and food industry, and the remaining effluent will have decreased phytotoxic properties, and thus it may be more safely used in agriculture.

Research on the agronomic effects of treated OMWW application to agricultural soil is limited. OMWW treated with activated charcoal and calcium hydroxide resulted in significantly improved seed germination of Italian ryegrass [15]. Increased productivity of maize and wheat was observed following the addition of OMWW processed, by liming and storage in a pond, and microbially, respectively [16, 17].

This paper describes the agronomic effects of 1 year application of treated OMWW, by microfiltration and resin, for polyphenols removal, to maize cultivation, with particular regards to crop yield and quality, and soil properties.

2. Materials and methods

2.1. Microfiltered olive mill wastewater (MF-OMWW)

A sample of approximately 10 Mg of OMWW was collected from “Alevizos” olive mill, located in Pyrgetos village, Larissa, central Greece. Initially, the raw OMWW was centrifuged at 1200 rpm using a rotary finisher bearing a stainless screen with holes of 150 μm diameter. As a second step, the treated OMWW was microfiltered using ceramic microfiltration membrane of 200 nm pore size to separate the polyphenols (permeate) from oil substances. Finally, the permeate produced in the second step was treated with the XAD4 macroporous resin, which has the ability to retain selectively the polyphenols [14], aiming to minimize any phytotoxic effects. Some quality properties of the produced MF-OMWW are presented in Table 1.

Table 1. Microfiltered olive mill wastewater quality properties.

Parameter	Value
pH	4.73
EC (mS cm^{-1})	9.91
TSS (%)	4.15
Available P (mg L^{-1})	1680
Extractable K (mg L^{-1})	1440
NH_4-N (mg L^{-1})	86

2.2. Field experiment

A field experiment was undertaken on a clay loam soil (41% sand, 20% silt, 39% clay) at the experimental farm of Technological Educational Institute of Larissa, Greece, in 2013. Topsoil quality properties are presented in Table 2.

Table 2. Topsoil properties at the beginning of the experiment.

Parameter	Value
pH	7.3
EC (mS cm^{-1})	0.723
Organic matter (%)	1.4
$CaCO_3$ (%)	1.8
Total N (%)	0.08
Olsen P (mg kg^{-1})	6,5
Extractable K (g kg^{-1})	0.468
Extractable Mg (g kg^{-1})	1.416

MF-OMWW was applied using two rates of 25 and 50 Mg ha^{-1}, with the addition of mineral fertilization of 200 kg N ha^{-1}. Furthermore, a treatment of only MF-OMWW applied at the rates of 50 Mg ha^{-1} and an only mineral fertilization treatment of 200 kg N ha^{-1} were used. Each treatment was applied to an individual plot of 60 m^2 (6 m x 10 m, including 8 plant rows), using a complete randomized block design with four replicates. Maize (*Zea mays*) was used as the monitoring crop. Crop sowing was at the rate of 8.6 seeds m^{-2}, and took place on May 25, 2013. To ensure germination, 30 mm was applied by sprinkler irrigation at sowing and an additional irrigation of 50 mm was applied later for seedling establishment.

Water and MF-OMWW were applied through a drip irrigation system, employing four manifolds. Each manifold supplied a set of four plots with one drip lateral per two adjusted plant rows. The volume of required water controlled by a flow meter installed at upstream of each manifold. The 20-mm diameter emitting pipe used, is commonly utilized for field crop irrigation, with pressure compensating emitters at 1 m spacing, discharging 3.6 liters per hour.

MF-OMWW was applied through the drip system utilizing a 120 L tank connected to the main line and manipulating a throttling valve to create a differential pressure level. Each treatment received water and MF-OMWW filtered through 1" conventional manual cleaning disk filters of 150 mesh. A preliminary 120 mesh screen filtration was operated on the main pipeline of the system. The secondary filters were cleaned after each MF-OMWW application. Manual flushing of the laterals was performed every third week. Five applications of MF-OMWW took place between 1 July and 8 of August delivering in total 1200 L for the two 50 Mg ha^{-1} MF-OMWW application rate treatments and 600 L for the 25 Mg ha^{-1} MF-OMWW application rate treatment.

An automatic weather station in the experimental field measured solar radiation, air temperature and humidity, and wind speed. These were used to calculate daily reference evaporation (ET_o). The irrigation applied through the drip system was scheduled using reference evaporation and growth stage based crop coefficient, according to FAO-56 methodology [18]. There was 108 mm of rainfall during the experiment. Total watering during the growing season was 500 mm with 310 mm applied through the drip system for all treatments. Fertilizer nitrogen was applied as ammonium nitrate using the fertilizer tank at a rate of 20 g N m^{-2}. All treatments were irrigated at 100% crop evapotranspiration (ET_c) during the full season.

Crop production was determined at harvest (September 20, 2013) by measuring fresh and dry weight of maize ears. Maize ears were harvested by hand from 10 maize plants from the central four rows of each experimental plot, when their dry matter content was on average 12.5%. Maize ears dry matter yield was determined by drying the ears in a ventilated oven at 60 °C, until constant weight. After drying, maize kernels were separated from the rest of the ear, grinded, and then analysed for protein, starch, ash and fiber content, using an automatic near infrared analyser.

Soil samples were collected three days after harvest. Samples were taken from 0-30 cm depth, from each plot. Soil samples were analysed for pH, EC, Olsen P, total N, extractable K, Ca, Mg, Na, NH_4-N and NO_3-N.

The effect of each treatment on crop and soil measured variables were assessed by ANOVA at the level of statistical significance of $p<0.05$, and means were separated by Duncan's test using the statistical program SPSS (Edit. 17.0).

3. Results and discussion

3.1. Crop production

Maize yield, which was determined by measuring the ears fresh mass, was not significantly affected by the different treatments as shown in Fig.1 (left). Average fresh and dry mass of maize ear are presented in Fig. 1 (right). Although the differences between the treatments are not statistically significant, there is a trend for better yield with the MF-OMWW application at the rate of 50 Mg ha^{-1}. This finding probably suggests that other nutrients contained in the MF-OMWW, rather than N, may have influenced crop production.

As far as maize kernel quality is concerned, kernel moisture and fat content were not significantly influenced by the different treatments, whereas kernel protein, starch, fiber and ash content were significantly affected (see Fig. 2 and 3). MF-OMWW application at the rate of 25 Mg ha^{-1} combined with mineral fertilizer N addition resulted in higher protein and ash content in comparison to the only MF-OMWW application. The later, however, resulted in higher starch content. Generally, in all treatments, kernel starch content responded in different manner than kernel protein and ash, which is in good agreement with other research work [19, 20]. Riedell [19] showed that kernel protein and mineral P and K content were higher, whereas starch content was lower, in plants grown under higher N input, which may also apply to the findings in our study.

Also, since the ear yield was about 20% higher for the only MF-OMWW treatment compared to the MF-OMWW treatment at the rate of 25 Mg ha^{-1} with the addition of mineral fertilizer N, the yield (on a kg ha^{-1} basis) of protein, starch and ash, is expected to be similar for the two treatments, which indicates that crop response to the different treatments was limited.

The important thing to note, however, is that considering all quality and quantity parameters studied, the treatment with only mineral fertilizer N application gave similar results with the only MF-OMWW treatment, indicating the potential of mineral fertilizer full substitution by MF-OMWW, under the conditions of our study.

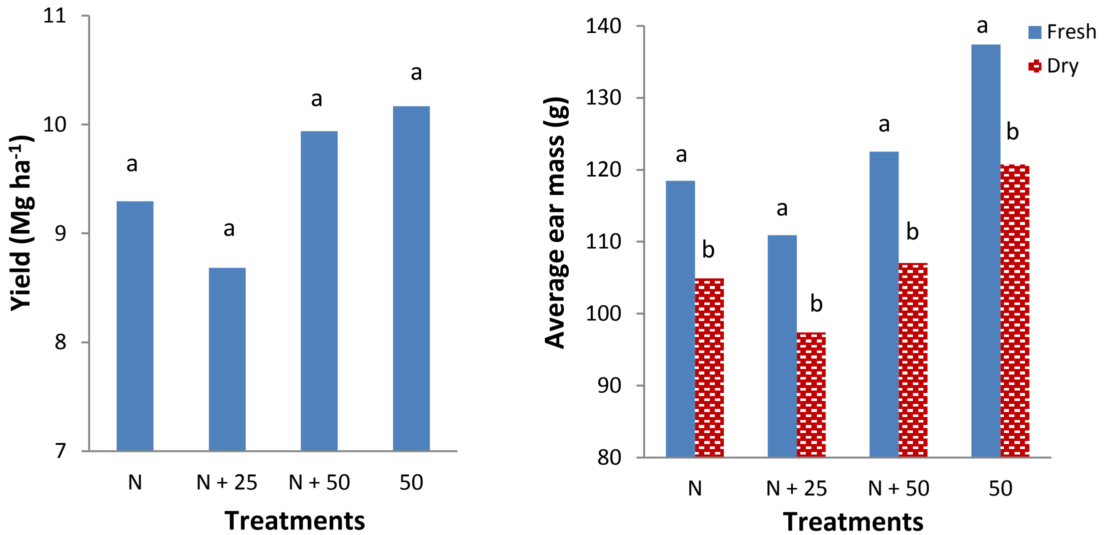

Fig.1.Maize yield (ears fresh weight) (left), and Average ear mass (fresh and dry) (right) for the different treatments (N: 200 kg N ha^{-1}, 25: 25 Mg MF-OMWW ha^{-1}, 50: 50 Mg MF-OMWW ha^{-1}). Columns labelled with the same letter are not significantly different ($P>0.05$).

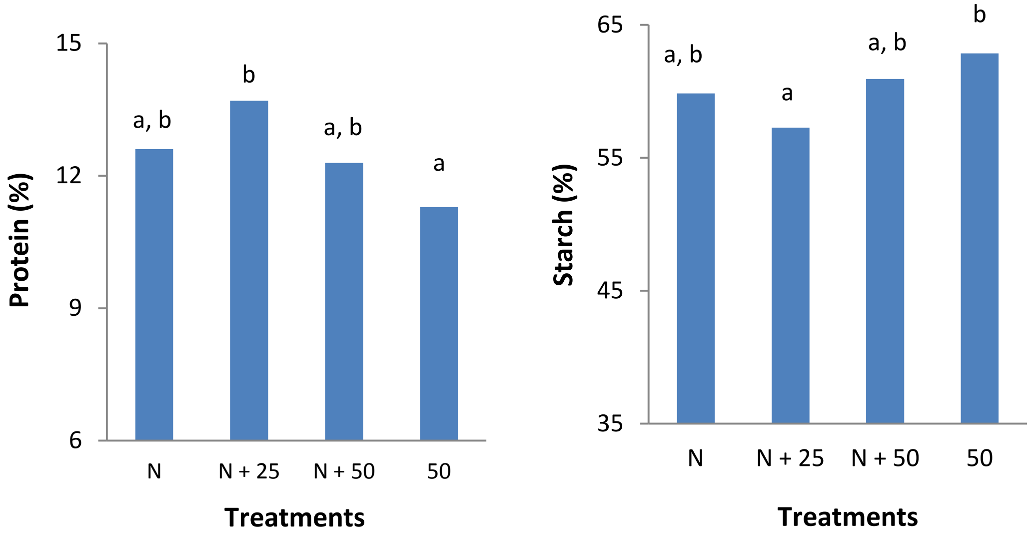

Fig. 2. Maize kernels protein (left) and starch (right) content for the different treatments (N: 200 kg N ha^{-1}, 25: 25 Mg MF-OMWW ha^{-1}, 50: 50 Mg MF-OMWW ha^{-1}). Columns labelled with the same letter are not significantly different (P>0.05).

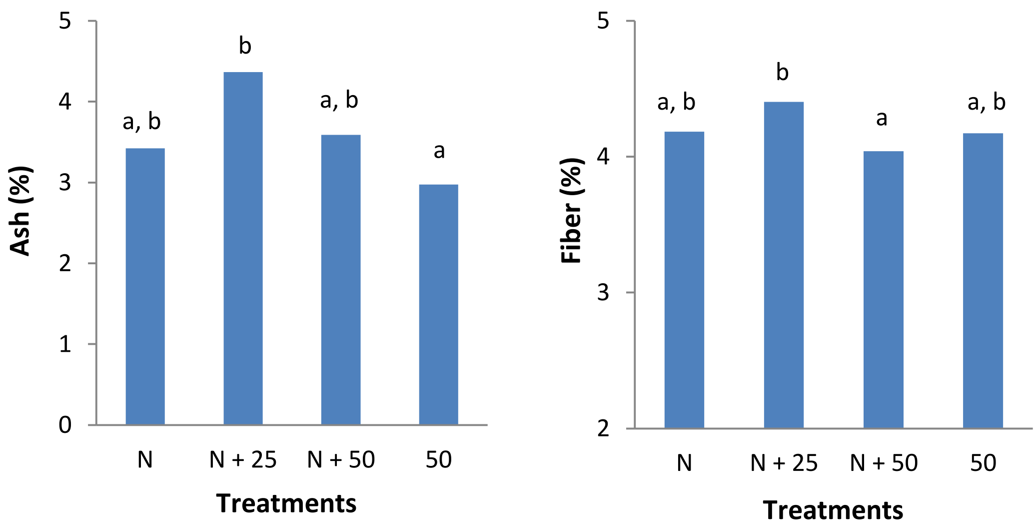

Fig. 3. Maize kernels ash (left) and fiber (right) content for the different treatments (N: 200 kg N ha^{-1}, 25: 25 Mg MF-OMWW ha^{-1}, 50: 50 Mg MF-OMWW ha^{-1}). Columns labelled with the same letter are not significantly different (P>0.05).

3.2. Soil properties

No significant differences between the different treatments were observed regarding all measured soil properties (pH, EC, available P, extractable K, Mg, Na, Ca, total N, and NO_3-N), with the exception of NH_4-N. Table 3 summarizes the results for the different treatments. The lack of soil response to the MF-OMWW application is mainly attributed to the relatively low rates of application used, and also to the fact that it was the first year of application. Other studies, which have shown increase in soil EC, P and N content following the application of treated OMWW (using other methods, e.g. lagoon stabilization), used higher rates of treated OMWW application [21, 22].

Table 3. Soil properties following one year MF-OMWW application to maize cultivation on clay loam soil. Mean values per treatment are only represented if statistically differences were found. In this case, numbers followed by different letter are statistically different.

Soil properties	Treatments (per ha)			
	50 Mg OMWW + 200 kg N	25 Mg OMWW + 200 kg N	50 Mg OMWW	200 kg N
pH	6.2			
EC (μS cm^{-1})	507.5			
P (mg kg^{-1})	5,9			
K (mg kg^{-1})	383,3			
Mg (mg kg^{-1})	1336,9			
Total N (%)	0.1			
NO_3-N (mg kg^{-1})	24.8			
NH_4-N (mg kg^{-1})	7.0b	4.7c	4.3c	9.7a
Na (mg kg^{-1})	110.9			
Ca (mg kg^{-1})	3008.7			

3.3. Economic analysis

Although crop and soil response to one year MF-OMWW application was limited, it was evident that MF-OMWW was capable to fully substitute the fertilizer N applied, under the conditions of our study. Considering that today the price of water soluble ammonium nitrate (which is the fertilizer used in this study) is about 0.46 € per kg, and the fact that this is a fertilizer commonly used by the farmers in the area at the rate of 200 kg N per ha, that leads to savings up to about 267 € per ha.

However, MF-OMWW transportation costs should also be taken into consideration. The preparation of MF-OMWW, as described in this paper, is not considered as an extra cost to the farmer, as it will be performed by the olive mill in order to reclaim the high added value polyphenols. As olive mills in Greece are scattered in different areas, for a transportation distance of around 50 km between the olive mill and the farm, an average transportation cost is around 5 € per Mg of MF-OMWW. Hence, for an application of 50 Mg MF-OMWW per ha, the cost accounts to 250 € per ha. In this case, farmers' savings are limited (only about 17 € per ha). However, the actual savings are expected to be higher following consecutive applications, due to potential soil amelioration.

Moreover, in another case scenario, the transportation costs may be charged to the olive mill. Other work has shown, that land application of OMWW at a maximum annual rate of 420 m^3 ha^{-1}, and considering an annual production of 1500 m^3 OMWW and a 10 year period of transportation equipment use (including the required investment costs: tractor and trailer with tank), cost less than 0.007€ per kg processed olive fruit [9].

4. Conclusions

The results of the 1st year of MF-OMWW application to maize production suggested that MF-OMWW could fully substitute mineral fertilizer N. Crop production was not enhanced by mineral fertilizer nitrogen addition to the MF-OMWW treated soil. Soil properties were not negatively affected by the application of MF-OMWWW. Considering the economics of MF-OMWW application, although there appears to be economic benefits to farmer from the mineral fertilizer substitution, the transportation costs need to be taken into consideration. Further work is necessary to evaluate the effects of MF-OMWW application to agricultural soil in the longer term.

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