

Maize Fertigation with Treated Olive Mill Wastewater: Effects on Crop Production and Soil Properties

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Abstract

The present study investigates the potential of olive mill wastewater, treated by microfiltration and XAD4 macroporous resin, to be used as liquid fertilizer in maize production through a 2-year field experiment. The treated olive mill wastewater (T-OMWW) was applied at two rates of 25 t and 50 t per ha per year, supplemented with mineral fertilization. There was also a treatment involving the application of only T-OMWW at the rate of 50 t per ha per year, and an only mineral fertilizer treatment. Mineral fertilizers and T-OMWW were applied progressively through a drip irrigation system.

Maize grain and soil analysis showed that T-OMWW was capable to meet crop requirements in N, P and K, and increase soil N, P and K availability. There was a tendency for increasing soil Na and electrical conductivity (EC) using the higher rate of T-OMWW. Therefore, for sustainable agriculture, it may be safer to apply the T-OMWW at the lower rate of 25 t per ha per year, or use the higher rate of 50 t per ha every other year.

Keywords: clay loam soil, kernel protein content, liquid fertilizer, microfiltration, XAD4 resin, yield

1. Introduction

Olive oil consumption is associated with many health benefits, including protective effects against cardiovascular diseases (Covas, 2007), cognitive decline (Berr et al., 2009) and possibly breast cancer (Escrich, Moral, & Solanas, 2011). The olive oil extraction process, however, involves the generation of large amounts of olive mill wastewater (OMWW), a by-product that constitutes serious environmental problem in the Mediterranean region, due to its high polluting load. OMWW is characterized by high content of solids and organic compounds, high COD content, phytotoxic properties and resistance to biodegradation caused by its phenolic compounds (Zirehpour, Jahanshahi, & Rahimpour, 2012; Zaglis, Vavouraki, Kornaros, & Paraskeva, 2013).

Olive oil production in Greece is mainly carried out by small or medium enterprises that usually apply the untreated OMWW to nearby land, in order to avoid treatment costs. Crop response to OMWW application is variable. Research has shown that olive fruit yield and quality were not affected by OMWW application (Chartzoulakis, Psarras, Moutsopoulou, & Stefanoudaki, 2010). Ryegrass and proteic pea yields were increased with untreated OMWW application, whereas clover yield was negatively affected (Montemurro, Diacono, Vitti, & Feri, 2011). In another study, although maize growth was not affected, plant stress parameters increased following the application of untreated OMWW (Belaqziz et al., 2008). Hanifi and El Hadrami (2008) found increased maize yield following moderate and progressive OMWW application. Germination problems are also observed due to phytotoxic effects of the phenolic compounds contained in the OMWW (Mekki, Dhouib, & Sayadi, 2007; Massoudinejad, Arman, & Aghayani, 2014). The application of untreated OMWW to agricultural soil may increase soil organic matter, available P and K (Montemurro et al., 2011), and total N content, but also soil electrical conductivity and salinity (Belaqziz, Lakhali, Mbouobda, & El Hadrami, 2008), and modify the equilibrium of useful soil microorganisms (Barbera, Maucieri, Cavallaro, Ioppolo, & Spagna, 2013).

The phenolic compounds contained in the OMWW are natural antioxidants, with commercial and economic interest. Hence, the treatment of OMWW aiming at the recovery of the polyphenols could result in economic

benefits for the olive mill, since the phytotoxic polyphenols will have been removed from the effluent. Membrane filtration of OMWW may result in a significant decrease of its organic load and suspended solids content (Russo, 2007, Zirehpour et al., 2012), and also in polyphenols separation from the mass of waste (Cassano, Conidi, & Drioli, 2011; Petrotos, Lellis, Kokkora, & Gkoutosidis, 2014; Rahmanian, Jafari, & Galanakis, 2014). OMWW treatment with microfiltration resulted in polyphenols separation in the permeate (Petrotos et al., 2014). Polyphenols may then be successfully removed with the use of suitable resins (Weisz, Schneider, Schweiggert, Kammerer, & Carle, 2010; Petrotos, Gkoutosidis, Kokkora, Giakidou, & Tsagkarelis, 2013; Zaglis et al., 2015). 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, and mainly involves OMWW that has been treated by chemical or biological techniques (Cereti et al., 2004, Barbera, Maucieri, Ioppolo, Milani, & Cavallaro, 2014; Moraetis, Stamati, Nikolaidis, & Kalogerakis, 2014). In this study, the effluent produced following the treatment of OMWW by microfiltration and XAD4 macroporous resin was applied by fertigation to maize production in a two-year field experiment. The aim of the study was to investigate its effects on crop production, with particular regards to grain yield and quality, and soil properties.

2. Method

2.1 Treated Olive Mill Wastewater (T-OMWW)

In 2013 and 2014, a sample of approximately 10 t of OMWW was collected each year from “Alevizos” olive mill, located in Pyrgetos village, Larissa, central Greece. Each year, the raw OMWW was initially centrifuged at 1200 rpm using a rotary finisher bearing a stainless screen with openings of 150 μm diameter. This first step aimed at separating the suspended solids contained in OMWW, in the form of sludge, in order to avoid clogging of the membrane used in the next step.

In the second step, the centrifuged OMWW was filtered using a ceramic microfiltration membrane of 200 nm pore size in order to separate the polyphenols in the permeate from the mass of waste (retentate). The effluent produced as permeate in this step was suitable to be applied through a drip irrigation system, with limited risk of emitters clogging.

As a final step, the permeate produced in the second step was passed through a column filled with XAD4 macroporous resin, which has the ability to retain selectively the polyphenols (Petrotos et al., 2013), aiming to recover the polyphenols and minimize any phytotoxic effects of the remaining effluent. The polyphenolic content of the remaining effluent was approximately 20-30% of the initial polyphenolic content of the input material.

The treated OMWW (the remaining effluent of the final stage) was considered for utilization in agriculture as a liquid fertilizer. Some quality properties of the treated OMWW (T-OMWW) are presented in Table 1 for each year. Table 1 shows that T-OMWW quality parameters varied between the two years, especially in respect of available nutrients. This variability is attributed to the annual variation of the nutritional quality of olives (input material) that may be processed by the olive mill each year, leading therefore to differences in the quality of OMWW and also T-OMWW.

Table 1. Treated OMWW physicochemical properties in 2013 and 2014

Parameter	2013	2014
pH	4.73	6.15
EC (mS cm^{-1})	9.91	8.30
Salinity (%)	8.9	6.0
Solid residue (105 °C) (%)	4.15	9.94
Available P (mg L^{-1})	1680	256
Extractable K (mg L^{-1})	1440	64
$\text{NH}_4\text{-N}$ (mg L^{-1})	86	3.11
$\text{NO}_3\text{-N}$ (mg L^{-1})	-	0.1

Note. EC: Electrical conductivity.

2.2 Field Experiment

A field experiment was carried out on an Inceptisol clay loam soil (41% sand, 20% silt, 39% clay) at the experimental farm of Technological Educational Institute of Thessaly, Larissa, Greece, in 2013 and 2014. Topsoil (0-0.3 m depth) quality properties in the beginning of the experiment are presented in Table 2.

Table 2. Topsoil (0-0.3 m depths) quality properties at the beginning of the experiment

pH	EC (mS cm ⁻¹)	O.M. (%)	CaCO ₃ (%)	Total N (g kg ⁻¹)	Olsen P (mg kg ⁻¹)	Extr. K (g kg ⁻¹)	Extr. Mg (g kg ⁻¹)
7.3	0.723	1.4	1.8	0.8	6.5	0.47	1.42

Note. EC: Electrical conductivity, O.M.: Organic Matter, Extr.: Extractable.

The experimental design involved four treatments: i) control (C): mineral fertilization only, ii) application of T-OMWW at the rate of 50 t ha⁻¹ (50W), iii) combined application of T-OMWW at the rate of 50 t ha⁻¹ with mineral fertilization (50W+f), and iv) combined application of T-OMWW at the rate of 25 t ha⁻¹ with mineral fertilization (25W+f). In 2013, the mineral fertilizer addition was equal to 200 kg N per ha for each of the three treatments that received mineral fertilizer. Fertilizer nitrogen was applied as ammonium nitrate (34.5-0-0). In 2014, the mineral fertilization for the control treatment corresponded to the addition of 110 kg N, 180 kg P₂O₅, 27 kg K₂O and 78 kg SO₃ per ha. The mineral fertilizer addition for each of the combined T-OMWW and mineral fertilizer treatments was 55 kg N, 131 kg P₂O₅, 27 kg K₂O and 17 kg SO₃ per ha. Three fertilizers were used in 2014: peckacid (0-60-20), urea phosphate (17.5-44-0) and ammonium sulfate (21-0-0). Each year, each treatment was applied to an individual plot of 60 m² (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 approximately 8.6 seeds m⁻², and took place on May 25, 2013 and June 18, 2014.

In order to ensure germination, sprinkler irrigation was applied each year after sowing for seedling establishment. Water, mineral fertilizers and T-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 L per hour.

Treated 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 T-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 T-OMWW application. Manual flushing of the laterals was performed every third week.

In 2013, five applications of T-OMWW took place between 1 July and 8 August, delivering in total 300 L for each plot receiving T-OMWW at the rate of 50 t ha⁻¹ and 150 L for each plot receiving T-OMWW at the rate of 25 t ha⁻¹. In 2014, six applications of T-OMWW took place between 1 August and 5 September, delivering in total 300 L for each plot receiving T-OMWW at the rate of 50 t ha⁻¹ and 150 L for each plot receiving T-OMWW at the rate of 25 t ha⁻¹.

All treatments were irrigated at 100% crop evapotranspiration (ET_c) during the full season, in both years of the experiment. An automatic weather station in the experimental field measured rainfall, solar radiation, air temperature and humidity, and wind speed. These parameters 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 (Allen, Pereira, Raes, & Smith, 1998). Table 3 shows monthly values of the measured parameters during the experimental periods. In 2013, there was 108 mm of rainfall during the experiment.

In 2013, there was 108 mm of rainfall during the experiment. Total watering during the growing season was 500 mm with 312 mm applied through the drip system for all treatments. In 2014, there was 96 mm of rainfall during the experiment, the majority of which took place after the irrigation period. Total watering during the growing season was 576 mm, with 480 mm applied through the drip system for all treatments.

Table 3. Monthly total rainfall, maximum (max) and minimum (min) temperature and humidity, average (aver.) wind speed and solar radiation during the two experimental periods

	Total rainfall		Max air temperature		Min air temperature		Max humidity		Min humidity		Aver. wind speed		Aver. solar radiation	
	(mm)		(°C)		(°C)		(%)		(%)		(m s ⁻¹)		(W m ⁻²)	
	2013	2014	2013	2014	2013	2014	2013	2014	2013	2014	2013	2014	2013	2014
Apr	2.4	75.2	31.6	25.2	2.4	0.4	100	100	19.0	24.9	1.5	1.5	519.6	430.5
May	56.4	10.2	34.8	33.2	8.5	5.5	100	100	11.5	13.2	1.7	1.4	557.3	567.3
Jun	72.6	23.4	38.4	40.0	9.0	10.9	100	100	7.8	11.7	1.5	1.4	616.5	597.7
Jul	50.2	12.2	38.9	38.0	14.2	14.5	100	100	19.6	18.5	1.2	1.7	632.9	648.5
Aug	0.0	8.8	38.7	40.4	14.6	13.5	99.5	100	14.0	16.2	1.4	1.5	596.7	594.9
Sept	13.0	81.6	36.5	34.1	8.8	7.5	100	100	13.2	26.0	1.3	1.3	478.4	451.1
Oct	15.0	85.8	27.9	29.6	3.8	5.5	100	100	13.3	28.8	0.8	0.8	311.4	234.2

2.3 Measurements and Analysis

Crop production was determined at harvest (September 20, 2013 and September 25, 2014). Maize ears were harvested by hand from 10 maize plants from the central 4 rows of each experimental plot. Maize ears were dried in a ventilated oven at 55 °C, until constant weight. After drying, maize kernels were separated from the rest of the ear, weighted, grinded, and then analyzed for protein, starch, fiber, oil and ash content, using an automatic near infrared analyzer. Reported kernel protein, starch, fiber, oil and ash content were corrected to 0% moisture content.

Soil samples were collected from each plot three days after harvest each year. Samples were taken from 0-30 cm depth and analyzed for pH (1:1 water), EC (1:1 water), Olsen P, Kheldahl N, extractable K, Na (determined in ammonium acetate extract by flame photometry), Ca, Mg (determined in ammonium acetate extract by atomic absorption), NH₄-N and NO₃-N (determined in KCl extract by phasmatophotometry).

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 multiple range test using the statistical program SPSS (SPSS Inc., Edit. 17.0, Chicago, USA).

3. Results and Discussion

3.1 Maize Kernel Yield and Quality

In both years of experimentation, the effect of T-OMWW application on maize kernel dry matter yield was not significant. As shown in Figure 1, however, in the second year there was a tendency for lower yield with the application of T-OMWW at the rate of 50 t per ha. This finding may be indicative of potential salinity effects on the crop. The higher salt concentration in the root zone resulting from the higher rate of T-OMWW application by drip irrigation may have contributed to the reduced yield, since maize is a salinity sensitive crop (Mahajan & Tuteja, 2005). Research work by Hanifi and El Hadrami (2008) showed variable toxicity levels depended on the salinity and phenolic content of olive mill wastewaters. They also showed that the application of OMWW at the rate of 30 m³ ha⁻¹ to maize production resulted in no physiological damage to the crop, which agrees with our results showing lack of adverse effects on yield with the lower application rate of T-OMWW (25 t ha⁻¹).

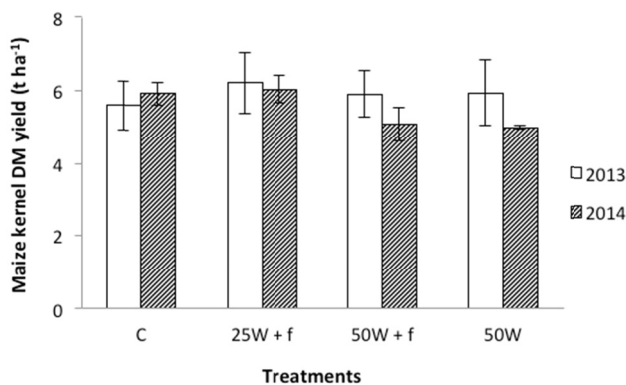


Figure 1. Maize kernel dry matter (DM) yield (in kg per ha) as affected by the different treatments, in the first and second year of the experiment. Error bars represent the standard error of the mean. (C: Control (mineral fertilization only), 25W + f: 25 t ha⁻¹ year⁻¹ of T-OMWW plus mineral fertilization, 50W + f: 50 t ha⁻¹ year⁻¹ of T-OMWW plus mineral fertilization, 50W: 50 t ha⁻¹ year⁻¹ of T-OMWW only)

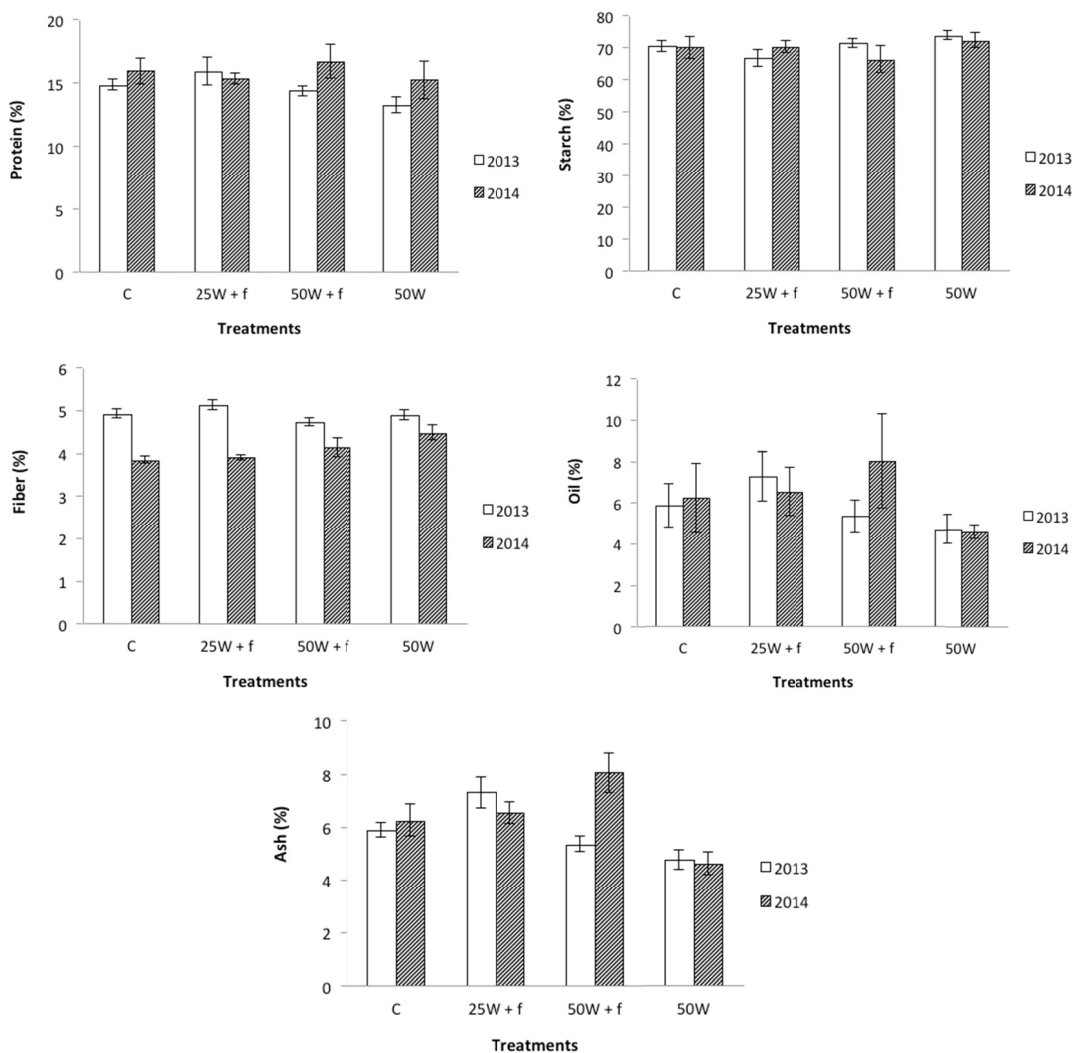


Figure 2. Maize kernel protein (top left), starch (top right), fiber (middle left), oil (middle right) and ash (bottom) content as affected by the different treatments, in the first and second year of the experiment. Error bars represent the standard error of the mean. (C: Control (mineral fertilization only), 25W + f: 25 t ha⁻¹ year⁻¹ of T-OMWW plus mineral fertilization, 50W + f: 50 t ha⁻¹ year⁻¹ of T-OMWW plus mineral fertilization, 50W: 50 t ha⁻¹ year⁻¹ of T-OMWW only)

Kernel quality parameters are presented in Figure 2. The application of T-OMWW, with or without the addition of mineral fertilization, resulted in similar content of protein with the control treatment in both years of the experiment. Mean kernel protein content was 15.2% (2-year average), which is a relatively high value. Kernel protein content generally increases with higher soil N availability (Oktem, Oktem, & Emeklier, 2010; Riedell, 2014), whereas N deficiency in soils affects negatively N metabolism and especially protein synthesis (Oktem, Ulger, & Kirtok, 2001). The similar protein content observed in all four treatments may suggest therefore that soil N availability for crop uptake was adequate to meet crop nitrogen requirements in all cases. This finding is very important as it indicates the potential of mineral fertilizer nitrogen full substitution by T-OMWW. Kernel starch, ash and oil content also ranged in similar levels with the control treatment in both years of the experiment. The application of T-OMWW at the rate of 50 t ha⁻¹ year⁻¹ resulted in significantly higher kernel fiber content in the second year of the experiment, in comparison to the other two treatments. These findings are indicative of T-OMWW capability to meet crop nutrient requirements and hence to be used as a liquid fertilizer.

In the second year of the study, kernel yield corresponding to the application of 50 t of T-OMWW per ha was about 15% lower than those of the control or the 25W+f treatment, corresponding to the combined application of T-OMWW at 25 t per ha and mineral fertilization (Figure 1). In order to further explore the potential impact of this non-significant trend in grain yield on kernel component concentrations, the total yield of kernel components on a kg ha⁻¹ basis were determined. Yields of protein, starch and fiber, and yields of oil and ash are presented in Figure 3, respectively, for both years of the experiment. There were no significant effects of the different treatments on yields of protein, starch, fiber, oil and ash.

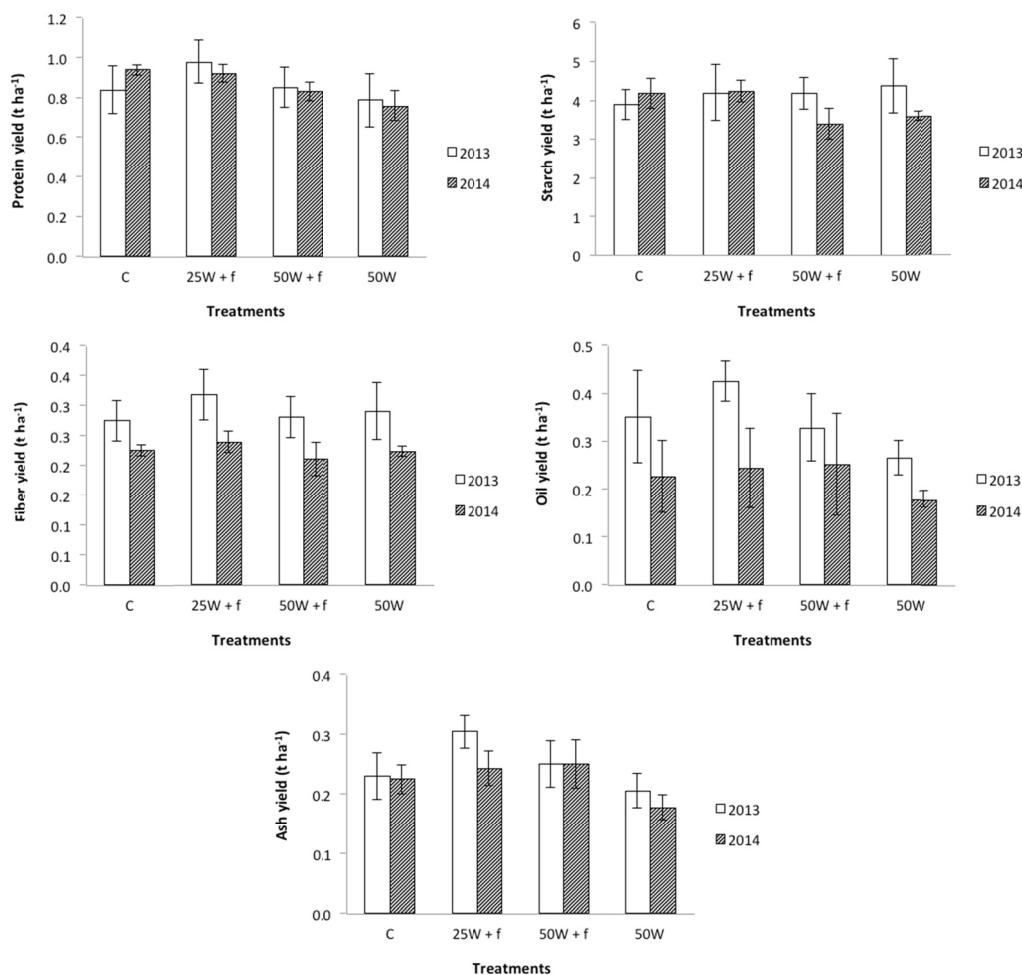


Figure 3. Maize kernel protein (top left), starch (top right), fiber (middle left), oil (middle right) and ash (bottom) yield (in kg per ha) as affected by the different treatments, in the first and second year of the experiment. Error bars represent the standard error of the mean. (C: Control (mineral fertilization only), 25W + f: 25 t ha⁻¹ year⁻¹ of T-OMWW plus mineral fertilization, 50W + f: 50 t ha⁻¹ year⁻¹ of T-OMWW plus mineral fertilization, 50W: 50 t ha⁻¹ year⁻¹ of T-OMWW only)

The results of T-OMWW application on maize grain yield and quality suggest that T-OMWW can be used as liquid fertilizer in maize production. The control treatment, corresponding to only mineral fertilizer application, gave similar results with the only T-OMWW treatment, indicating the potential of mineral fertilizer substitution by T-OMWW, under the conditions of our study. Although not clearly shown within the first two years of maize fertigation with T-OMWW, it seems that there is an advance in grain yield with applying the T-OMWW at the lower rate of 25 t per ha.

3.2 Soil Properties

The effect of T-OMWW application on soil properties was not obvious following the first year of application (results can be found in Kokkora et al., 2015). The second year of application gave a clearer picture of the effects of T-OMWW on soil properties, although again the differences between the treatments were not significant.

As shown in Table 4, there was a trend for gradual increase in soil EC following the application of greater quantities of T-OMWW. Other research work has also shown increase in the EC of a clayloam soil after the implementation of OMWW processed in stabilizing tanks (Siera, Martin, Garau, & Cruañosm, 2007). This observation implies that caution is required in continuous application of T-OMWW to soils with high clay content, due to potential increase in salts concentration, and/or salinity sensitive crops. Soil sodium content also seemed to increase with T-OMWW application (Table 3). Magdich et al. (2013) observed significant increase in soil Na content following the successive 3-year application of OMWW at rates higher than 50 m³ ha⁻¹ to sandy soil. Due to the fact that our soil was rich in Mg and Ca (mean values at the end of the growing season was approximately 1436 and 3213 mg kg⁻¹, respectively), sodicity problems were not an issue in this soil. Problems may arise, however, with the continuous application of T-OMWW in the case of soils poor in total salts and/or sodium sensitive crops.

Soil extractable K was practically unaffected by the different treatments (Table 4), although there was a slight tendency to increase by combining T-OMWW and inorganic K fertilization. At the end of the first growing season, soil extractable K was at the level of approximately 383 mg kg⁻¹, while at the end of the second growing season was at least 516 mg kg⁻¹. This finding indicates that the application of T-OMWW only, with no inorganic K addition, was capable to meet crop K requirements and also to increase soil K availability.

Soil available P content increased with the application of mineral fertilizer P and the combination of T-OMWW with reduced mineral fertilizer P addition. Our soil was poor in available phosphorus and the mineral fertilizer P addition in the control treatment aimed both at meeting crop P requirements and also enriching soil P levels. Indeed, the mineral P fertilization met crop requirements and increased soil P levels by approximately 41% in comparison to the soil P levels at the end of the first growing season. The combination of reduced mineral fertilizer P addition and T-OMWW further increased soil P levels (approximately 55% increase compared to control soil P levels at the end of the first growing season). The application of only T-OMWW slightly increased soil P levels by about 7%. These results indicate the potential of T-OMWW to increase soil available P levels. In the case, however, of soils poor in P, the combined application of T-OMWW and mineral fertilizer P seems as the most favorable option.

Soil O.M. content was not affected by T-OMWW application (mean soil O.M. content was 1.2% within the top 30 cm). Mean soil pH was 6.6. As for soil nitrogen, mean total N content was 1.0 g kg⁻¹, the residual nitrate-N content was 29.8 mg kg⁻¹ and the residual ammonium-N content was negligible (less than 2.5 mg kg⁻¹) for all treatments. Soil residual mineral N corresponds to approximately 148 kg nitrate-N per ha. This is a relatively high value and may lead to increased nitrate leaching losses during winter. T-OMWW application at the rate of 50 t per ha was capable to meet crop N requirements and increase soil N availability, which suggests therefore that the extra fertilizer N added with the treatment of 50W+f (combining T-OMWW applied at 50 t per ha along with mineral fertilizer N), possibly leached below the top 30 cm of soil during the growing season, hence posing a serious threat to the environment. It is evident from these results that no addition of mineral fertilizer N was necessary with the application of 50 t of T-OMWW per ha.

Table 4. Indicative topsoil (0-0.3 m depth) properties following two years T-OMWW application by drip irrigation to maize cultivation on clay loam soil. (C: Control (mineral fertilization only), 25W + f: 25 t ha⁻¹ year⁻¹ of T-OMWW plus mineral fertilization, 50W + f: 50 t ha⁻¹ year⁻¹ of T-OMWW plus mineral fertilization, 50W: 50 t ha⁻¹ year⁻¹ of T-OMWW only)

	EC (mS cm ⁻¹)	Na (mg kg ⁻¹)	K (mg kg ⁻¹)	P Olsen (mg kg ⁻¹)
C	0.5 ^a (0.06)	158.0 ^a (6.06)	524.3 ^a (9.18)	8.3 ^a (1.85)
25W + f	0.6 ^a (0.06)	228.0 ^a (19.63)	555.7 ^a (5.31)	9.0 ^a (1.15)
50W + f	0.8 ^a (0.17)	194.3 ^a (47.28)	540.0 ^a (31.58)	9.3 ^a (1.21)
50W	0.7 ^a (0.23)	240.3 ^a (2.71)	516.7 ^a (13.57)	6.3 ^a (0.87)

Note. Numbers in brackets are standard error of mean. Columns labeled with the same lower case letter are not significantly different ($P > 0.05$).

EC: Electrical conductivity.

4. Conclusions

The results of the present study showed the utilization potential of treated olive mill wastewater, by microfiltration and XAD4 resin, as liquid fertilizer in maize production. Plant and soil analysis showed that T-OMWW was capable to meet crop requirements in N, P and K, and also to increase soil N, P and K availability. In a soil poor in available P, the combined application of T-OMWW and fertilizer P was necessary to substantially increase soil available P levels. A non-significant tendency for increasing soil Na and EC levels and reducing grain yield was observed with the higher rate of T-OMWW application of 50 t per ha. These results indicate that for sustainable agriculture, it may be safer to apply the T-OMWW at the lower rate of 25 t per ha per year, or to consider the application of 50 t per ha every other year. Further research work is necessary to evaluate the long-term effects of T-OMWW application on soil quality and crop production.

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