

## **THE IMPACT OF OLIVE MILL WASTEWATER ON THE PHYSICO-CHEMICAL AND BIOLOGICAL PROPERTIES OF SOILS IN NORTHWEST JORDAN**

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### **Abstract**

Soil contamination may influence negatively soil health, which often limits and sometimes disqualifies soil biodiversity and decreases plant growth. Soil health is the continued capacity of the soil to function as a vital living system, providing essential ecosystem services. Within soils, all bio-geo-chemical processes of the different ecosystem components are combined. These processes are able to sustain biological productivity of soil, to maintain the quality of surrounding air and water environments, as well as to promote plant, animal, and human health. A common criterion to evaluate long term sustainability of ecosystems is to assess the quality of soil. However, the increased concentration and distribution of toxic substances in soils by mismanagement of industrial activities, overuse of agrochemicals and waste disposal are causing worldwide concern. A major environmental concern in the Mediterranean countries is the production of the large quantities of olive oil mill wastewater (OMW) produced during olive oil extraction process. OMW inhibits several groups of bacteria and fungal species, thus affecting soil stability. In the present study, we investigated the effect of OMW on the soil physical, chemical characteristics and the microarthropods structure. All soil samples were collected from an olive mill garden in Northwest Jordan. Biological soil quality index (QBS-ar) values appeared to decrease with respect to soil pollution by OMW. All investigated parameters were significantly different depending on the levels of OMW contamination in soil. Anthropogenic activities influenced the microarthropod community, altering both quantity and quality of soil chemical and physical structure of the microhabitats. Preliminary data obtained in this study suggest that the application of QBS-ar index could be a useful tool for evaluating surface soils health status.

**Keywords:** *olive oil mill wastewater, heavy metals, soil biological quality*

### **Introduction**

Soil is a key resource for the life of organisms in terrestrial environments; it is the basis of the ecosystem and of our farming system of food production. Soil contamination may influence negatively soil health, which often limits and sometimes disqualifies soil biodiversity and decreases plant growth. Soil health is

the continued capacity of the soil to function as a vital living system, providing essential ecosystem services (Wahsha et al., 2012a; Bini, 2007). Within soils, all bio-geo-chemical processes of the different ecosystem components are combined. These processes are able to sustain biological productivity of soil, to maintain the quality of surrounding air and water environments, as well as to promote plant, animal, and human health. A common criterion to evaluate long term sustainability of ecosystems is to assess the quality of soil. However, the increased concentration and distribution of toxic substances in soils by mismanagement of industrial activities, overuse of agrochemicals and waste disposal are causing worldwide concern (Wahsha and Al-Rshaidat, 2014). A major environmental concern in the Mediterranean countries is the production of the large quantities of olive oil mill wastewater (OMW) produced during olive oil extraction process (Lozano-García and Parras-Alcántara, 2013). The average annual production of olive oil is about 3 million tons worldwide and more than 98% of the world's olive oil production takes place in Mediterranean region, whereas Spain produces 43% of world production of olive oil (Justino et al., 2009). Jordan is the 8<sup>th</sup> largest olive oil producing country in the world; it has more than 15 million olive trees that produce over 130,000 tones of olives every year. Seventy per cent of the mills are located in the northern region, 22 per cent in the central region and 8 per cent in the south (Jordan times, 2014). OMW has an offensive smell, dark red to black color and high concentrations of organic contents, polyphenols, polyalcohols and lipids. If it spreads on soil or is dumped in wadis, OMW can cause serious environmental problems and reduce soil fertility as it contains many chemicals, with a strong toxicity to microorganisms and plants (Deeb et al., 2012). Reports showed that OMW inhibits several groups of bacteria and fungal species, thus affecting soil stability (Ochando-Pulido et al., 2012). Despite being recognized as a truly hazard residue it is still often being discharged onto soil or into water courses, especially by small oil mills, causing severe environmental impacts (Gámiz et al., 2012). Moreover, OMW toxic properties have been investigated for their harmful effects in plants, animals and bacteria. Recently, several reports investigated the effect of OMW on the biota and the results have been shown that OMW can play a role in physiological alterations both at the organism and cellular levels (Justino et al., 2009). Several studies were performed aiming to assess the impact of OMW on the fertility of soils, chemical properties and plant performances of cultivated lands (Mekki et al., 2013; Gámiz et al., 2012; Ochando-Pulido et al., 2012). The underestimation of key factors, such as soil texture and properties, moisture, water table, type of crop and the uncontrolled spreading showed to produce negative impacts on soil plant relationship (Barbera et al., 2013). However, moderate and controlled OMW land application may result in no negative long-term effects on soil chemical parameters and in improving soil fertility and productivity. So far in Jordan, little interest has been given to the impact of OMW land spreading on soil properties. In this study we investigated the

effect of OMW contaminated soils on the soil biological, physical and chemical characteristics from soils in Northwest Jordan.

## **Materials and methods**

### **Site description**

The soil samples were collected from an olive oil factory farm (2000 m<sup>2</sup>) in Northwest Jordan. The area is consisting of mountains that are famous for their lush vegetation and thick green forests and a good place for hikes. Its highest mountain peaks reach around 1268 meters above sea level and Ajlun mountains receive a few snow storms every year usually in winter season from December to March. Ajlun has a Mediterranean weather rainy and snowy in winter season and pleasant in the summer time.

### **Samples collection**

Seven representative sites were selected according to homologous geological, morphological and pedological conditions, vegetation coverage and anthropogenic impact and the same pedoclimate conditions. The distance between sampling sites ranged from 20 to 25 m. Soil pits were opened and samples were collected from the upper horizon at a depth of 0-30 cm for routine analysis, and 0-15 cm (O+A horizons) for QBS-ar test. Successively, all locations were sampled for topsoils in the period of spring 2014. According to the procedures described by Margesin and Schinner (2005), soil samples were air dried at room temperature for 7 days, homogenized and sieved through a stainless-steel sieve of 2 mm mesh diameter before the determination of heavy metals, physico-chemical soil properties and quantification of soil microbial communities structure.

### **Laboratory analysis**

On the fine soil part, routine analyses (bulk density, pH, carbonate content, organic carbon and texture) were carried in order to define basic soil characteristics. pH in 1:2.5 soil: water suspension using glass electrode (Violante and Adamo, 2000); total carbonates (gas-volumetric method); organic carbon by the Walkley and Black (1934), Soil particle size distribution was determined by pipette method according to Genevini et al., 1994. For the analysis of pseudo-total metal content in soils, 0.2 g of powder soil sample was subjected to a complete digestion in 5 mL of *aqua regia* in the microwave (Milestone Ethos EZ) in closed containers made of Teflon. After digestion, soil samples were analyzed by flame atomic absorption spectrometry. The quality control procedures consist of 70 measuring soil double replicates and six standard certified reference materials purchased from Inorganic Venture Spain (M)SDS. For QBS-ar evaluation, soil samples were carefully placed on the mesh filter (2 mm) above the Berlese-Tullgren funnel for 14 days. Soil microarthropods were collected and identified by light microscope and each type of organisms found in every sample was evaluated according to its adaptation to soil edaphic environment according to Parisi et al. (2005).

## **Results and Discussion**

The area of consideration in this study, located on Ajlun municipality, is an important olive oil producer site in Jordan. It is therefore an extremely important resource for the local economy income. Our findings with regard to the production of olive oil mill wastewater showed that the area is highly contaminated by several different potentially toxic materials, as will be discussed later on. Soils were classified as Vertisols according to Soil Survey Staff (1999). In particular, Xererts are the Vertisols developed under a xeric soil moisture regime. They are clayey soils, developed on hard limestone and basalt. These soils have a high water holding capacity because of their high clay content. The physical properties of the studied area are greatly influenced by moisture content. The bulk density varies between 0.5-2.2 g/cm<sup>3</sup> because of their swelling and shrinking nature with changes in soil moisture content. The soils have high bulk density when these are dry and low values when in a swollen stage. Soil pH ranged between 7.3 and 8.6 in the soil profile due to the presence of CaCO<sub>3</sub> and high contents of bases, especially calcium and magnesium in the soil. Organic matter has been found to be more or less uniformly distributed, varies from 30 to 35 g Kg<sup>-1</sup>. Table 1 summarizes the results of the average concentrations of Cr, Cu, Fe, Mn, Pb and Zn in the soils tested. The total concentrations of most of the investigated metals (Cu, Fe, Pb and Zn) in the soil samples were significantly higher (ANOVA P < 0.05) than those of normal range in control soils, and (Cr, Cu, Pb and Zn) were above the toxicity threshold according to Canadian thresholds legislation for agricultural soils. All heavy metals concentrations recorded at the investigated site do not overcome the international industrial threshold limits as reported by Wahsha et al. (2012a). Cr, Cu, Pb and Zn are above the critical soil total concentration range in control soils (Alloway, 1995), the range of values of the investigated metals are above the level where toxicity for the environment is considered to be possible.

**Table 1.** Concentration of heavy metals in soils from an olive mill garden in Northwest Jordan. Data are expressed as mg kg<sup>-1</sup>. \*All the values are mean of seven replicates ± S.D.

Element	1 Con **	2 Crit. Con **	3 OMW ***	4 Inv. Sam *	5 Can. thr ***	6 IL **
Cr	200	75-100	0.15± 0.05	70 ± 16	64	800
Cu	20	60-125	2.31± 0.07	100 ± 11	63	600
Fe	1000	-	6.41± 0.13	1730 ± 35	-	-
Mn	850	1500-3000	4.23± 0.10	556 ± 14	-	-
Pb	10	100-400	-	180 ± 29	70	1000
Zn	50	70-400	3.83± 0.17	137 ± 21	200	1500

1 Con = Maximum normal range in soils; 2 Crit. Con = Critical soil total concentration;

3 OMW = Olive oil mill wastewater; 4 Inv. Sam = Investigated samples;

5 Can thr = Canadian thresholds for agricultural soils; 6 IL = Industrial Limits.

(\* adopted from Wahsha et al., 2012 and \*\*\* from Komnitsas et al., 2011).

The linear correlation (Table 2) between Pb, Cu, Zn (Cu/Pb 0.593; Pb/Zn 0.687; Cu/Zn 0.611) significant at ( $P < 0.05$ ) is consistent with their calcophilous behavior, since these metals tend to form compounds with sulfur, as chalcopyrite ( $\text{CuFeS}_2$ ), sphalerite ( $\text{ZnS}$ ) and galena ( $\text{PbS}$ ). Cr is negatively correlated with Cu ( $-0.521$ ), Pb ( $-0.403$ ) and Zn ( $-0.529$ ). Fe and Mn are not significantly correlated with any other element, although they share the same geochemical behavior, as a result of anthropogenic activities in the area (Wahaha et al., 2014). It is noteworthy to point out, however, that the OMW levels of contamination in the investigated area vary proportionally with the heavy metals in soils of the corresponding site.

	Cr	Cu	Fe	Mn	Pb	Zn
Cr	1					
Cu	-0.521	1				
Fe	0.094	0.074	1			
Mn	0.120	0.032	0.184	1		
Pb	-0.403	0.593	0.265	0.065	1	
Zn	-0.529	0.611	0.182	0.174	0.687	1

**Table 2**  
*Linear correlation coefficients calculated on the concentrations of metals in soils. Indicated correlation is significant at the 0.05 level (2-tailed).*

The QBS-ar values fall within a wide range (between 50 and 80) and indicates a state of suffering of the soil due to an environmental stress. According to Parisi et al. (2005), QBS-ar values between 100 and 200 identify a stable ecosystem with good quality. The microflora could be affected by the change in the soil chemical conditions and the heavy metals. Some authors reported that some residual polyphenolic compounds presents in OMW are toxic for this sensitive category of microarthropods (Wahsha and Al-Rshaidat, 2014).

There was a significant difference in the microbial community between soils affected by OMW ( $P < 0.05$ ) and those of control, as it was observed by calculating the coefficient of determination ( $R^2$ ) between the variables considered.  $R^2$  explains how much of the OMW contaminant level receive to soils is correlated to the microbial biomass in soils and it was in our study ( $R^2 = -0.756$ ), indicating a negative relationship between OMW levels in soils and the microbial biomass, confirming the anthropogenic impact of OMW on the environment. The soil microarthropods respond much more quickly than most other soil fractions to changing environmental conditions, such as variations in geogenic inputs from the substrate input or increases in pollutant content. However, the presence of contaminants (such as OMW and heavy metals) in the soil can influence the microbial metabolism processes by affecting the enzyme activities. OMW may reduce the enzyme activities by masking catalytically active groups (ROS production), having denaturing effects on the conformation of proteins, or competing with the metal ions involved in the formation of enzyme substrate complexes. Wahsha et al. (2013) showed that the microbial activity is a much more

sensitive indicator of changing soil conditions soil conditions than other physical and chemical indicators.

Thus both activity and biomass can serve as an early warning of such changes, long before they are detectable in other ways. The presence of *Acarina*, *Symphyla*, *Protura* and *Collembola* is important, being considered metal-tolerant (Wahsha et al., 2012b): for example, *Symphyla* seem to be quite affected by high lead concentrations and our results show a decrease in their abundance in areas with high concentrations of Pb, Zn and Cu. There is now a considerable amount of literature to show that soil microarthropods measurements are useful in determining effects of stresses on the soil ecosystem.

## **Conclusions**

The addition of unprocessed OMW can causes significant shifts that might negatively reduce the structure and function of soil. The application of QBS-ar index can be used as a tool for assessing toxic elements alterations, before severe disturbances such as population changes occur, thus providing a detailed picture of the soils' health and the status of the surrounding environment.

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