

**OLIVE OIL ELEMENTAL CONTENT OF TWO LOCAL GENOTYPES AS
INFLUENCED BY RECYCLED EFFLUENT IRRIGATION
UNDER ARID ENVIRONMENT**

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ABSTRACT

Effect of recycled effluent irrigation was investigated on quality, elemental composition (Ca, Cd, Co, Fe, K, Mn, Mg, Na, Pb Cl and Zn) and of two local olive genotypes (*Olea europaea* L.) under field conditions during two complete vegetative cycles. The analysis of the effluent indicated that element concentrations fall within the permissible range for irrigation except Na and Mg. The Ca, Cl, K, Mn, Na and Zn were found in all oils of trees irrigated with wastewater and fresh water. However, the concentration of these elements was higher in the olive oil of trees irrigated with treated wastewater than olive oil of trees irrigated with fresh water but lower than the maximum permissible concentration. The concentrations ranged from 8.90 to 26.15 mg kg⁻¹ for Ca; 0.07 to 0.13 % for Cl; 53.19 to 111.75 mg kg⁻¹ for K, 0.97 to 1.46 mg kg⁻¹ for Mn; 6.24 to 16.10 mg kg⁻¹ for Na and 0.19 to 0.36 mg kg⁻¹ for Zn. The effect of irrigation water quality on oil quality indices (peroxide and acidity) was not significant. There were significant correlations between the concentrations of the pairs Ca-Na, Zn. These results indicate that this kind of effluent is suitable for irrigation of olive genotypes grown for oil purposes.

Key word: irrigation, minerals, oil quality, olive, recycled water.

1. INTRODUCTION

Water scarcity in the Mediterranean countries, especially in countries in the arid zone with high rates of population growth, urbanization and industrialization, appears as one of the main factors limiting agricultural development. Jordan is considered to be one of the ten poorest countries worldwide in water resources. Due to the limited water resources and the relatively high population growth rate (2.5 %, 2004), the annual per capita share is expected to decrease from 160 m³ in recent years to less than 90 m³ by 2025 (Shatanawi *et al.*, 2003). The irrigation share of the total water uses demonstrates significant decrease during the period 1985-2008 (78% in 1975 to 60 % in the year 2008). Within the next 25 years, although irrigated areas will increase, large quantities of fresh water supplies will be diverted from agriculture to meet the growing water demand in the municipal and industrial sectors in the region (Hamdi *et al.*, 1995; Correia, 1999). In order to overcome water

shortages and to satisfy the increasing water demand for agricultural development, the use of water of low quality (brackish, reclaimed, drainage) is becoming important in many countries like Jordan.

An alternative plan for saving water could include the use of non-conventional water resources such as the treated wastewater. The main health risks are associated with contamination of crops by wastewater due to its chemical composition being somewhat different from most natural waters used in irrigation (Pereira *et al.*, 2002). Ion toxicity and nutritional disturbances are detrimental effects of wastewater on plant growth and yield quality (Cheesman, 1988). Siebe (1995) found that wastewater irrigation increased heavy metals contents of soils with time, and plant uptake of Cd and Pb also increased.

Olive (*Olea europaea* L.) has been grown commercially in Jordan for many years. In the last three decades, olive plantation extended almost to all regions of the country, particularly the eastern and northern parts.

Currently, olive plantations encompass about 103, 840 ha with annual production of around 148.2 thousand tons (Ministry of Agriculture, 2005). The objective of this work was to study the effects of the reuse of recycled effluent (from Al-Hussein Bin Abdullah II Industrial Estate Wastewater Treatment Plant) as textile factories wastes mixed with municipal domestic effluent on olive oil quality of two local olive cultivars (about 70% of the cultivars currently grown in Jordan) at field conditions.

2. MATERIALS AND METHODS

2.1. Experimental sites and water source

This study was carried out at the olive orchard of Al-Hussein Bin Abdullah II Industrial Estate (HUIE) which is located about 20 km east of Karak city during the period March, 2004 - January, 2006. The area is characterized with cool winter and very hot summers. The total rainfall in average years is below 150 mm/ year. The treated wastewater used in this study was taken from the HUIE Wastewater Treatment Plant. This water is mainly generated by textile firms mixed with municipal domestic effluent. The treatment current flow is about 500 m³ d⁻¹. Chemical characteristics of the effluent were determined during the growing season as presented in (Table 1).

2.2. Orchard site and irrigation treatments

Five years old trees of the two cultivars ('Nabali' and 'Improved Nabali') were used at the field experiment. These genotypes were selected for their widespread planting in orchards and nurseries, both private and public. The olive trees in the field experiment were trickle irrigated with fresh water (av. EC 0.82 dS m⁻¹) and treated wastewater (av. EC 4.20 dS m⁻¹). Both treatments were given the same amount of irrigation quantity. In order to define water volumes, ET_c (crop evapotranspiration) was calculated multiplying ET_o by crop coefficient (K_c ranged from 0.65 to 0.70). The total irrigation quantity applied each season was about 800 mm. The fruits were harvested from olives trees from both treatments and oil was extracted. Oil elemental composition and quality were analyzed.

2.3. Oil microwave-assisted acid decomposition

Oil samples were stored in dark glass bottles at 4°C until the time of analysis. To

dissolve the oil samples for elemental analysis, microwave-assisted acid decomposition was performed at high pressure and temperature. The digestion was carried out as follows: 6 ml of nitric acid at 69.5%, and 1 ml of hydrogen peroxide 30% were added to 1 g of oil. A CEM model MarsX Microwave-assisted system was used. The microwave operation parameters were: power 250 W for 1 min, 0 W for 1 min, 250 W for 5 min, 400 W for 5 min, 650 w for 5 min; maximum temperature was 200 °C. After digestion, samples were allowed to cool to room temperature for 12 hr; the residues were taken up to 25 ml with bi-distilled water.

2.4. Oil elemental composition determination

The Ca, Cd, Co, Cu, Fe, K, Mn, Mg, Na, Ni, P, Pb and Zn concentrations were determined by atomic emission spectrometer (Jobin Yvon Emission - JY 38 S, Paris, France) (Tandon, 1995). Detection limits for the elements were below 0.1 mg kg⁻¹. The calibration was performed by using five aqueous standard solutions in 5% HNO₃ (v/v). The calibration standard solutions have the following concentrations: 0, 0.2, 0.5, 2.0, and 5.0 mg/L. The acidity of the standard and sample solutions was the same (5% HNO₃). Chloride was titrated by AgNO₃.

2.5. Statistical design and analysis

Randomized complete block design (RCBD) consisted of two cultivars, two irrigation water qualities and four replicates were used at HUIE field experiment. Each replicate was represented by three trees. Statistical assessments of differences between mean values were performed by LSD test at P=0.05 according to Snedecor and Cochran (1980) using MSTATC statistical package (Michigan State Univ., East Lansing, MI).

3. RESULTS AND DISCUSSION

3.1. Irrigation water quality

The chemical analysis of the treated wastewater and fresh water used for irrigation was determined by the methods described by Greenberg *et al.* (1985) (Table 1). In general, levels of most metals were below the recommended maximum concentrations and within guidelines for irrigation of agricultural crops (Ayers and Westcot, 1985). It is clear that the wastewater had higher concentrations of sodium (Na) and magnesium (Mg) than

Table (1): Chemical characteristic of the treated waste water and potable used for irrigation.

Parameter	Mean ± St. dev			Parameter	Mean ± St. dev		
	WW	FW	CLWW		WW	FW	CLWW
pH	7.8 ± 0.30	7.60 ± 0.17	8.00	Cl (mg L ⁻¹)	93.20 ± 23.69	82.00 ± 8.82	350.00
NO ₃ -N (mg L ⁻¹)	3.90 ± 1.42	3.40 ± 0.23	30.00	Fe (mg L ⁻¹)	0.72 ± 0.13	0.10 ± 0.02	5.00
NH ₄ -N (mg L ⁻¹)	4.10 ± 2.07	2.10 ± 0.12	10.00	Mn (mg L ⁻¹)	0.06 ± 0.01	nd	0.20
PO ₄ (mg L ⁻¹)	8.60 ± 1.39	0.08 ± 0.01		Zn (mg L ⁻¹)	0.04 ± 0.00	0.10 ± 0.01	2.00
K (mg L ⁻¹)	22.40 ± 6.01	6.75 ± 0.67		Cu (mg L ⁻¹)	0.09 ± 0.03	0.01 ± 0.00	0.20
Ca (mg L ⁻¹)	99.03 ± 31.20	51.80 ± 7.02	400.00	Pb (mg L ⁻¹)	0.02 ± 0.00	nd	5.00
Mg (mg L ⁻¹)	84.19 ± 17.92	28.30 ± 3.39	60.00	Ni (mg L ⁻¹)	0.01 ± 0.00	0.10 ± 0.01	0.20
Na (mg L ⁻¹)	275.25 ± 55.22	50.70 ± 8.05	207.00	Cd (mg L ⁻¹)	nd	nd	0.01

CLww: Concentration limits of irrigation water; WW: Treated wastewater; FW: Fresh water; St. dev.: Standard deviation; nd: Not detected.

Table (2): Chemical and physical characteristics of the soil in the experiment site at beginning of the experiment.

Soil properties	Mean	Soil properties	Mean
Texture	SCL	Na (mg kg ⁻¹)	2.10
pH	8.10	Fe (mg kg ⁻¹)	4.90
EC (dS m ⁻¹)	1.40	Mn (mg kg ⁻¹)	8.00
OM (%)	0.81	Zn (mg kg ⁻¹)	3.20
N (%)	0.22	Cu (mg kg ⁻¹)	2.50
P (mg kg ⁻¹)	224.00	Pb (mg kg ⁻¹)	0.24
K (mg kg ⁻¹)	154.00	Ni (mg kg ⁻¹)	nd
Ca (mg kg ⁻¹)	326.00	Cd (mg kg ⁻¹)	nd
Mg (mg kg ⁻¹)	37.70		

SCL: Silty clay loamy soil; OM: Organic matter.

the recommended maximum limit (Ayers and Westcot, 1985). On the contrary, the levels of calcium (Ca), copper (Cu), iron (Fe), manganese (Mn), nickel (Ni), lead (Pb), and zinc (Zn) were within the sufficiency range for plant growth. The level of cadmium (Cd) was below the detection limit.

3.2. Soil properties

Some properties of the soil used in this study are given in Table (2). The soil texture is sandy clay loam. It is clear that the soil tend to have relatively high pH value (1:1). Soil analyses have shown that soil has low E.C. (1.4 dS m⁻¹, 1:1) and low organic matter content (0.81%). The total soil-N was between 0.15% - 0.22%, while the available P was between 155-224 mg kg⁻¹ which is considered optimal for most crops (Walsh and Beaton, 1973). Trace and micro elements concentrations were relatively low.

3.3. Olive oil elemental compositions

The main effects of wastewater irrigation on the elemental composition of olive oil are divided into three groups of elements: (1) trace elements (Cd, Co and Pb,) and salts (Cl and Na); (2) micro nutrients (Fe, Mn and Zn);

and (3) macronutrients (Ca, K and Mg).

3.3.1. Trace elements

The trace elements (Cd, Co and Pb) and salts (Cl and Na) are not essential for plantnutrition in large amounts, and can have phytotoxic effects, even at low concentrations. Among heavy metals, Cd plays a major role; its presence is due to the growing use of sewage sledges and other wastes in agricultural lands. Cd is absorbed by plants and enters the food chain; and may cause severe problems to human health (Crosby, 1977). However, the concentration of Cd in oil was undetectable for both cultivars and irrigation treatments. The Co and Pb were undetectable in the olive oil of both cultivars. Increasing salinity of wastewater significantly increased olive oil Cl contents of 'Improved Nabali' and 'Nabali' oils (Fig. 1a). This result is highly confirmed by many authors (Al-Gazzaz, 1999; Bernstein *et al.*, 2006; Al-Absi *et al.*, 2008) wastewater increased Cl content in the aerial part of the olive tree. Results of Cl and Na accumulation in 'Nabali' oil support previous work (Tattini *et al.*, 1992; Klein *et al.*, 1994) describing ion exclusion and retention of Cl as well as Na in aerial part of the olive tree.

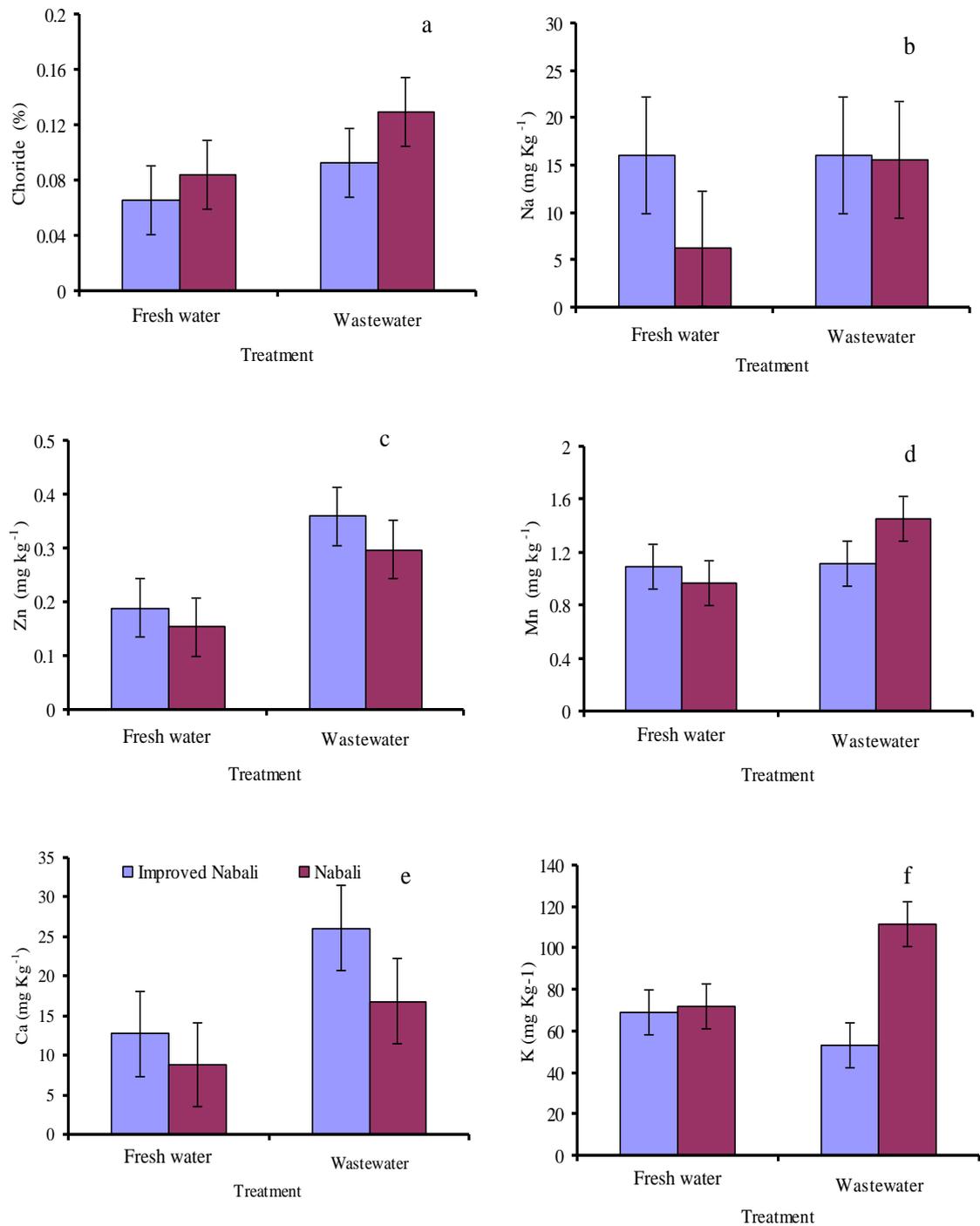


Fig. (1): Olive oil contents of chloride (a), sodium (b), zinc (c), manganese (d), calcium(e) and potassium (f) of 'Improved Nabali' and 'Nabali' olives as influenced by recycled effluent irrigation.

The Na concentration of 'Nabali' olive oil increased using wastewater irrigation; however, 'Improved Nabali' shows no effect (Fig. 1b). Even though, the contents of leaf Na of the two cultivars were increased (Al-Absi *et al.*, 2008). This can be explained to higher exclusion and translocation of Na to the upper parts of the olive tree in 'Nabali' for salt tolerance. Tolerance of olive tress to salt seemsto be cultivar-dependent (Chartzoulakis, *et al.*, 2004). At low and moderate salinities leval, most olive cultivars exhibit Na exclusion capacity (Tattini *et al.*, 1992; Chartzoulakis *et al.*, 2002). A relatively positive high correlations between the concentrations of the pairs of Na and Ca ($r=0.77$), Zn ($r=0.43$) were noticed.

3.3.2. Micro nutrients

Although essential for plant growth, micronutrients (Fe, Mn and Zn) can produce toxic effects if they reach very high concentration in the plant tissue.

in the soil.

Despite the clear effect of increasing Zn accumulation induced by wastewater irrigation (Fig. 1c), olive oil content of Zn was relatively far from the maximum permissible concentration for oil (10 mg kg^{-1}) (Angelova *et al.*, 2004). There were a relatively positive correlations between the concentrations of the pairs of Zn and the other elemental (Ca ($r=0.68$), Na ($r=0.43$)).

3.3.3. Macro nutrients

Irrigation with treated wastewater relatively increased the oil content of Ca, K and Mg (Figs.1e and f). A considerable difference was found in the elemental composition between the two cultivars. It is clear in Fig. (1e) that increasing salinity of wastewater increased the oil Ca content for both cultivars. There were a relatively positive high correlations between the concentrations of the pairs of Ca and Na ($r=0.77$), Zn ($r=0.68$).

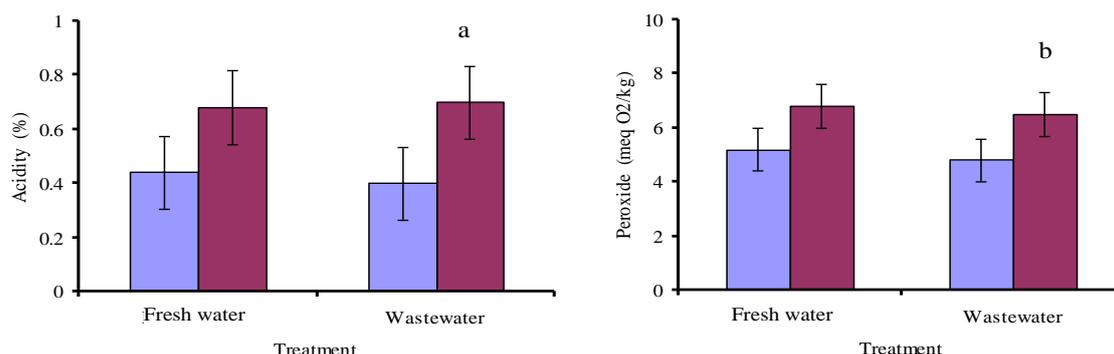


Fig. (2): Olive oil acidity percentages(a) and peroxide values (b) of 'Improved Nabali', and 'Nabali' olives as influenced by recycled effluent irrigation.

Concentrations of Fe in olive oil were undetectable and lower than the accepted maximum permissible concentrations for Fe (0.1 mg kg^{-1}) (Angelova *et al.*, 2004). The results showed that irrigation of 'Nabali' with treated wastewater significantly increased the oil content of Mn, while the oil Mn content of oil of 'Improved Nabali' remained constant (Fig.1d). Oil Mn contents of both cultivars under irrigation with both fresh and treated wastewater were within the range accepted for human health (WHO). The concentration of Mn in wastewater was relatively low and the accumulation of Mn found in olive oil for both cultivars and treatments, must be a reflection of an inherent high Mn availability

A considerable variation in the oil K content between the two tested cultivars irrigated with treated wastewater was observed. 'Nabali' had significantly higher K oil content than 'Improved Nabali' (Fig. 1f). The observed increase in K concentration at the current investigation, which resulted in a relatively similar K/Na ratio, may provide a mechanism by which olive plant achieve ionic balance following uptake of Na. The analysis indicated presence of a relatively positive weak correlation between oil Na and K concentrations. A relatively positive and negative weak correlation between the concentrations of the pairs of K and other oil elemental content of Ca, Na, Zn, and Mn.

The Mg concentration was undetectable in oils of both cultivars and irrigation treatments.

3.4. Oil quality

Peroxide values and acidity percentages were not significantly affected by irrigation with treated wastewater (Fig.2a and b). The oxidation occurrence as a result of mechanical injuries to fruit accrued during harvest, which detrimental to oil quality, leads to increased acidity and peroxide values (Servili *et al.*, 1999). In our study, the fruit was picked carefully by hand so that no injuries were inflicted; peroxide and acidity were not changed due to irrigation with wastewater compared with fresh water (Fig.2). This makes sense since it is well known that these indices of are mostly affected by fruit handling and paste manipulation (Uceda and Hermoso, 1999) rather than source of irrigation.

Conclusion

Regarding olive oil elemental composition and quality, it seems that recycled effluent can be used for olive trees irrigation as the elemental concentrations were lower than the maximum permissible concentration and the oil quality indices were not changed using treated wastewater. Metallic cations: Cd, Co, Fe, Mg, Pb were below the detected limits and below the maximum permissible concentration. Ca, K Mn, Na, Zn were found in all oils. The concentrations of these elemental were relatively higher in the olive oils irrigated with treated wastewater but lower than the maximum permissible concentration. There were significant correlations between the concentrations of the some pairs of elemental. These results indicate that this kind of wastewater is suitable for application to olive orchards under irrigation for olive oil production purposes.

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تأثير الري بالمياه المعالجة على نوعية الزيت لصنفين من أصناف الزيتون المحلية تحت الظروف الجافة

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ملخص

تم دراسة تأثير الري بالمياه المعالجة على نوعية الزيت والمحتوى المعدني (Ca, Cd, Co, K, Fe, Mg, Mn, Na, Pb, Zn) لصنفين من أصناف الزيتون المحلية تحت ظروف الحقل خلال دورتي نمو خضري. أظهر تحليل المياه المعالجة أن تركيز العناصر يقع ضمن المدى المسموح به لمياه الري عدا تراكيز الصوديوم و المغنيسيوم. دلت النتائج على وجود تراكيز معتبرة من Mn, Zn, Ca, Na, K, Cl في زيت الزيتون المروي بالمياه المعالجة و المياه العذبة. كما وجد أن تركيز هذه العناصر في زيت الزيتون المروي بالمياه المعالجة كانت أعلى نسبياً من تركيز العناصر في زيت الزيتون المروي بالمياه العذبة إلا أنها كانت أقل من الحد الأعلى المسموح به صحياً. تراوحت تراكيز هذه العناصر من 8.90 - 26.15 ملغم/كغم للكالسيوم، 0.07 - 0.13 % للكلور ، 53.19 - 111.75 ملغم/كغم للبتوتاسيوم، 0.97 - 1.46 ملغم/كغم للمغنيز، 6.24 - 16.10 ملغم/كغم للصوديوم، 0.19 - 0.36 ملغم/كغم للخارصين، أما بالنسبة لمعياري نوعية زيت الزيتون (النسبة المئوية للحموضة و رقم التزرنخ) فلم تتأثر معنوياً بنوعية مياه الري. أظهرت النتائج وجود ارتباط معنوي بين بعض العناصر المقاسة Ca-Na, Zn. تبين النتائج صلاحية استخدام هذه النوعية من المياه المعالجة لري أشجار الزيتون بهدف الحصول على الزيت.

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