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EFFECTS OF IRRIGATION WITH WASTEWATER ON LEVELS OF MACRO ELEMENTS AND HEAVY METALS IN VALENCIA ORANGE TREES GROWN IN DESERT SOIL:

I- UPTAKE AND PARTITIONING DURING FRUIT DEVELOPMENT

Mahdy, A. M.*; D. O. El-Ansary[1] and A. M. Hussein****

*** Department of Soil and Water Sciences, Faculty of Agriculture (El-Shatby), Alexandria University, Alexandria, 21545, Egypt**

**** Department of Pomology, Faculty of Agriculture (El-Shatby), Alexandria University, Alexandria, 21545, Egypt**

[1]Corresponding author: Dr. D. O. El-Ansary; E-mail: diaaagri @ hotmail. com

ABSTRACT

The objectives of this research were to investigate the soil pollution load and to determine the effects of irrigation with wastewater on macro elements and heavy metals uptake and partitioning in Valencia orange trees [*Citrus sinensis* (L.) Osbeck] during the active fruit development stage in the desert of west Noubaria. The results indicated that the application of wastewater led to changes in some soil physicochemical characteristics. Macro elements (N, P, and K) and heavy metals (Cd, Mn, Cu, Ni, Pb, and Zn) concentrations in wastewater-irrigated soil were higher than the concentrations in the canal water-irrigated reference soil. Concentrations of heavy metals in wastewater-irrigated soil, except for Mn, were above the maximum allowed levels for soils. The pollution load index values indicated that the wastewater-irrigated soil was strongly enriched with heavy metals. Wastewater-irrigated trees were contaminated with heavy metals and the levels in developing fruits exceeded the maximum allowed daily intake (MADI) for human. The trend of plant concentration factor (PCF) for heavy metals in roots, leaves and fruits was in the order of Zn> Cd > Ni > Pb> Mn > Cu in wastewater-irrigated trees, and was Cd > Mn > Pb > Cu > Ni > Zn in canal water-irrigated trees.

INTRODUCTION

Orange is the most important fruit crop grown in Egypt, with approximately half the total fruit production in Egypt. Orange cultivation in Egypt is concentrated in two main regions, the Nile Delta and the newly reclaimed desert lands. However, in the desert of west Nubaria, there is an increasing demand for irrigation water resources. Therefore, some farms reuse the drainage water or wastewater to fulfill the irrigation requirements especially during summer season. Some villages in west Noubaria dispose untreated municipal water in drainage water canals and therefore the drainage water is mixed with untreated municipal water. This mixed wastewater is used by some growers to irrigate their farms during the whole year where river Nile canal water cannot reach or satisfy the optimum irrigation needs and groundwater from wells is expensive to be used. Wastewater irrigation, solid waste disposal, sludge applications, vehicular exhaust and industrial activities are the major sources of soil contamination with heavy metals, and an increased metal uptake by food crops grown on such contaminated soils is often observed. In general, wastewater contains substantial amounts of beneficial nutrients and toxic heavy metals, which are creating opportunities and problems for agricultural production, respectively (Chen *et al.*, 2005 and Singh *et al.*, 2004). Excessive accumulation of heavy metals in agricultural soils through wastewater irrigation, may not only result in soil contamination, but also lead to elevated heavy metal uptake by crops, and thus affect food quality and safety (Muchuweti *et al.*, 2006). Heavy metal accumulation in soils and plants is of increasing concern because of the potential human health risks. Food chain contamination is one of the important pathways for the entry of these toxic pollutants into the human body. Heavy metal accumulation in plants depends upon plant species and the efficiency of different plants in absorbing metals, and this can be evaluated by either plant uptake or soil-to-plant transfer factors of the metals (Rattan *et al.*, 2005). Fruits cultivated in wastewater-irrigated soils take up heavy metals in large quantities which may lead to potential health risks to the consumers. In order to assess the health risks, it is necessary to identify the potential of a source to introduce risk agents into the environment, estimate the amount of risk agents that come into contact with the human-environment boundaries, and quantify the health consequence of the exposure (Ma *et al.*, 2006). According to the National Research Council (NRC, 1983), this process consists of four steps, hazard identification, exposure assessment, dose/response assessment, and risk characterization. Chronic level intake of toxic metals has adverse impacts on humans and the associated harmful impacts become apparent only after several years of exposure (Bahemuka and Mubofu, 1999 and Ikeda *et al.*, 2000). However, the consumption of heavy metal-contaminated food can seriously deplete some essential nutrients in the body that are further responsible for decreasing immunological defenses, intrauterine growth retardation, impaired psycho-social faculties, disabilities associated with malnutrition and high prevalence of upper gastrointestinal cancer rates (Iyengar and Nair, 2000 and Turkdogan *et al.*, 2003). Wastewater irrigation is a widespread practice in the world and recently a number of articles have been published on wastewater-irrigated soils contaminated with heavy metals (Liu *et al.*, 2005; Mapanda *et al.*, 2005; Rattan *et al.*, 2005 and Rothenberg *et al.*, 2007). However, an additional insight into metal uptake, accumulation and assessment of human health risks associated with wastewater-irrigated soils is still needed. In Egypt, there are increasing environmental and public health awareness regarding the pollution of soil, water, and fruits as a result of using untreated wastewater in agriculture. The objectives of this research were to investigate the soil pollution load and to determine the effects of irrigation with wastewater on macro elements and heavy metals uptake and partitioning in Valencia orange trees grown on the desert soils of west Noubaria.

MATERIALS AND METHODS

Plant materials and growing conditions: The study was conducted during the growing seasons 2008 and 2009 at two private Citrus orchards located in Tiba region in west Noubaria, Egypt. The first site (long. 29.54 °E, lat. 30.41 °N) was irrigated with canal water from river Nile and consisted of 5 feddans of four-year-old Valencia orange trees [*Citrus sinensis* (L.) Osbeck] grafted on

Volkamer lemon rootstock [*Citrus limon* Burm f.], and the trees were planted in single rows spaced 4 m within rows and 5 m between rows. The second site (long. 29.56 °E, lat. 30.36 °N) was irrigated with mixed drainage and municipal wastewater and consisted of 100 feddans of four-year-old Valencia orange trees grafted on Volkamer lemon rootstock, and the trees were planted in single rows spaced 4 m within rows and 6 m between rows. The majority of citrus tree roots were in the top soil layer (30 to 60 cm). Irrigation was delivered using two drip irrigation tubes per row. Fertigation was applied once a week as adapted in the area. Each feddan received 120 : 60 : 150 units of N, P, and K, respectively during the season. Soil, water, root, leaf and fruit sampling was conducted in the mid of June in 2008 and 2009 growing seasons.

Soil sampling and analysis: Surface (0-30 cm) and sub-surface (30-60cm) soil samples (*Typic torripsamment*) were collected using a stainless steel auger. Samples of the air-dried soils were ground to pass a 2-mm sieve prior to the following physical and chemical analysis: Soil pH was measured in 1:2.5 soil suspension (Richards, 1954) and electrical conductivity (EC) was determined using the paste extract method (Richards, 1954); Calcium carbonate content was determined by using a calcimeter (Nelson, 1982); Particle size distribution was measured according to the hydrometer method (Day, 1965); The organic matter content (OM) of the samples was determined by dichromate oxidation method (Nelson and Sommers, 1982); Cation exchange capacity (CEC) was determined using 1 M NaOAC (Rhoades, 1982). In addition, the available macronutrients; Olsen-P (Olsen and Sommers, 1982); NH_4OAc-K (Knudsen *et al.*, 1982) and available-N (Keeney and Nelson, 1982) were determined. The properties of the studied soil in the two seasons are presented in Table (1). Total heavy metals (Cd, Cu, Ni, Pb, Mn, and Zn) were determined according to Ure (1995). The diethylene triamine penta acetic acid extracting (0.005M DTPA, 0.1 TEA, and 0.01 M $CaCl_2$, adjusted to pH 7.3) solution (Lindsay and Norvell, 1978) was employed to extract heavy metals as potential indicator of plant-available heavy metals from soils irrigated with and without mixed water. The soil extracts were analyzed for the determination of Cd, Cu, Ni, and Pb by the atomic absorption spectrometry (Baker and Amacher, 1982).The total and DTPA-extractable heavy metals content in soil for the two seasons are shown in Table (2).

Water sampling and analysis: Representative water samples (500 ml) were collected in polyethylene bottles, which properly washed/rinsed with the same water that is being sampled. Water samples were taken from two sources, the first source represents canal water (Fara Eshreen canal) from Nile river, and the second represents wastewater (Fara Eshreen drainage) where drainage water is mixed with municipal wastewater. After proper labeling (e.g. source of water, date of collection, and type of analysis required), the samples were sent immediately to the laboratory.

Table (1): The main Physical and chemical properties of the studied soil (means ± SD). #

Treatment	Depth	EC	pH	$CaCO_3$	CEC,	Clay	Silt	Sand	Texture¶	Available-K (1N NH_4 OAc-K)	Available-P (0.5 N $NaHCO_3$)	Available –N (2M KCl)	O.M†
	cm	dSm^{-1}		gkg^{-1}	Cmol(+).kg^{-1}	gkg^{-1}				$mgkg^{-1}$			gkg^{-1}
	1st season												
Canal water	0-30	0.62±0.03	7.38±0.03	22.5±0.12	9.65±0.21	111.00±6.00	24.45±0.55	858.24±4.76	L.S	72.00±3.10	2.89±0.14	49.00±1.40	11.00±2.00
	30-60	0.44±0.02	7.42±0.02	36.5±0.23	8.99±0.23	115.00±3.00	27±0.50	870±5.00	L.S	68.00±1.60	1.89±0.11	46.00±2.00	8.90±0.30
Wastewater	0-30	3.25±0.04	7.82±0.03	32.5±1.30	7.55±0.20	111.98±5.45	24.98±0.72	858.96±5.04	L.S	85.60±0.40	5.27±0.05	58.00±0.20	14.00±1.00
	30-60	3.45±0.06	7.44±0.02	34.50±1.50	8.21±0.09	115.50±2.55	27.30±0.45	871.6±3.8	L.S	84.00±2.00	4.56±0.43	55.00±1.70	12.30±0.40
LSD 0.05		0.05	0.03	2.10	0.25	6.10	0.75	6.22		2.69	0.31	1.98	1.50
	2nd season												
Canal water	0-30	0.66±0.02	7.48±0.02	23.00±0.12	10.00±0.23	113.00±4.53	22.34±3.50	864.66±2.55	L.S	76.12±2.54	3.34±0.09	51.65±0.98	15.12±1.54
	30-60	0.41±0.04	7.35±0.03	37.45±0.06	9.60±0.09	118.12±6.12	26.45±3.50	855.43±4.33	L.S	67.98±0.99	2.11±0.06	49.09±1.54	11.54±0.23
Wastewater	0-30	3.67±0.07	7.83±0.04	33.30±0.07	8.86±0.05	114.12±5.50	23.56±0.56	862.32±4.87	L.S	91.12±0.54	7.23±0.06	60.11±0.15	18.99±0.88
	30-60	4.00±0.11	7.50±0.03	35.65±0.03	9.12±0.31	117.09±2.43	25.13±0.98	857.78±2.99	L.S	88.85±0.35	6.60±0.33	58.98±0.89	15.76±0.43
LSD 0.05		0.05	0.03	1.99	0.23	6.13	0.73	6.17		2.23	0.28	1.95	1.51

Means of three samples ± SD.

† O.M: organic matter

¶ L.S: Loamy sand

Table (2): The amount of total and DTPA-extractable heavy metals contents in soil irrigated with canal and waste waters (means ± SD). #

Treatment	Depth,cm	Total heavy metals						DTPA-extractable metals					
		mg.kg^{-1}											
		1st season											
		Cd	Cu	Pb	Ni	Mn	Zn	Cd	Cu	Pb	Ni	Mn	Zn
Canal water	0-30	2.10±0.11	43.21±0.22	234.00±6.00	14.00±0.11	89.12±2.12	185.00±2.00	0.18±0.02	3.13±0.05	2.18±0.08	5.13±0.05	1.55±0.07	12.24±0.26
	30-60	1.86±0.11	44.50±1.50	254.00±6.00	16.12±0.46	95.00±2.00	179.00±2.00	0.33±0.06	2.45±0.09	1.89±0.03	4.76±0.21	1.23±0.12	15.54±3.00
Wastewater	0-30	7.13±0.31	112.22±2.22	912.00±4.00	56.67±2.67	276.00±3.00	523.54±4.46	3.12±0.06	34.65±1.35	55.54±1.46	18.76±0.24	27.16±0.26	55.65±2.33
	30-60	7.02±0.13	115.00±2.57	899.00±11.00	55.50±1.50	295.00±4.00	530.34±11.66	2.99±0.13	36.87±3.87	60.34±1.66	17.65±1.05	31.67±1.33	53.00±2.43
LSD $_{0.05}$		0.24	2.47	9.62	2.06	3.85	8.53	0.10	2.72	1.35	0.73	0.90	3.00
		2nd season											
Canal water	0-30	2.20±0.21	45.21±2.22	238.00±6.42	18.00±1.56	92.12±2.73	189.00±3.05	0.22±0.06	5.22±0.04	3.18±0.92	7.13±1.95	3.55±1.09	15.24±1.51
	30-60	3.30±0.66	48.50±2.75	254.00±6.00	19.50±2.00	99.00±3.05	182.00±2.64	0.38±0.06	4.45±1.23	2.22±0.19	5.76±0.61	4.23±1.45	17.54±1.52
Wastewater	0-30	9.13±0.31	118.22±4.11	918.00±5.00	61.50±1.50	276.90±3.04	533.54±7.30	4.12±0.63	38.65±2.67	59.54±2.90	22.76±2.32	29.16±1.18	59.65±3.28
	30-60	8.02±0.87	121.00±1.78	902.00±2.00	60.00±2.00	285.00±7.02	530.34±11.66	3.99±0.59	37.87±2.39	63.34±2.40	19.70±1.58	34.67±2.18	58.40±3.95
LSD $_{0.05}$		0.22	2.45	9.58	2.03	3.78	8.48	0.10	2.71	1.33	0.72	0.89	3.02

The water samples were filtered and analyzed for EC, soluble cations and anions (Richards,1954), but the pH of water samples was measured before filtration of samples (Richards,1954); Sodium adsorption ratio(SAR) was calculated in order to determine the sodicity or alkalinity hazard of irrigation waters; NO_3-N was determined by the Kjeldahl method (Keeney and Nelson, 1982); Boron was determined colorimetrically using carmine method (Richards,1954); and soluble heavy metals were determined spectrophotometrically using atomic absorption spectrometry (AAS) (Baker and Amacher, 1982). Water analysis data are presented in Table (3)

Plant sampling and analysis: Each sample replicate of newly developed roots consisted of two sub-samples that were taken from both sides of one tree under the dripper at the active root-zone (0 - 60 cm deep). Each sample of leaves consisted of 30 mature leaves from the middle of non-fruiting shoots from all directions of the tree. Each fruit sample consisted of 10 developing fruits of approximately 5 cm in diameter that was collected from all directions of the tree. The plant tissues were washed with tap water followed by double-distilled water, dried in a forced-air oven at 65 °C for 48 h, and ground in a stainless steel mill. Ground plant samples were ashed in a muffle furnace at 450 °C for 6 h (Jones, 2001). The plant ash was dissolved in nitric acid solution (1:1, v/v), diluted to a final volume with double-distilled water, and analyzed for Cd, Pb, Cu, Zn, Mn, Ni, K and P. Another sub-sample of plant material was ashed and dissolved in hydrochloric acid solution (1:1, v/v), diluted to a final volume with double-distilled water, and analyzed for N (Jones, 2001).

Plant concentration factor and soil pollution load index calculations:

1- **Plant concentration factor (PCF):** The transfer factor of heavy metals from soil to plant tissues was calculated on the basis of dry weight as follows: $PCF= C_{plant} / C_{soil}$

Where C_{plant} and C_{soil} represent the concentrations of heavy metals in plant tissue and available concentrations in soil, respectively, (Cui *et al.*, 2005).

2- **Pollution load index (PLI):** The degree of soil pollution for each metal was calculated using the following modified equation:

$$PLI= C_{sample\ soil} / C_{reference\ soil}$$

Where $C_{sample\ soil}$ and $C_{reference\ soil}$ represent the concentrations of heavy metals in wastewater-irrigated soil and canal water-irrigated soil, respectively, (Liu *et al.*, 2005).

Statistical analysis: Three and five representative random replicates for soils and plant tissues, respectively, were sampled for both treatments in each season. Concentrations of macro elements and heavy metals in plant tissues were analyzed by the t-test. Soil characteristics for both treatments in two soil layers were analyzed by one-way analysis of variance (ANOVA) and significance was determined by the Fisher's least significant difference test at $P< 0.05$ (SAS Institute, 1994).

Table (3): The chemical analysis of the irrigation waters used in the study (means ± SD except for pH). #

Irrigation	EC	pH	Cl^{-1}	Na^{+1}	Ca^{+2}	Mg^{+2}	SAR	NO$^{-1}_{3}$-N	HCO$_3^{-1}$	B	Cu^{+2}	Pb^{+2}	Ni^{+2}	Mn^{+2}	Zn^{+2}	Cd^{+2}
	dSm^{-1}		meq.l^{-1}							mg.l^{-1}						
	1st season															
Canal water	0.62± 0.05	8.17- 8.21	9.00± 0.70	3.70± 0.10	3.60± 0.50	0.40± 0.03	1.90± 0.13	4.00± 0.50	4.00± 0.20	0.60± 0.04	0.18± 0.02	3.20± 0.09	0.11± 0.03	0.14± 0.02	1.30± 0.19	0.02± 0.01
Waste water	4.86± 0.07	7.89- 7.97	25.00± 1.10	24.50± 0.50	15.00± 1.70	10.00± 0.50	5.50± 1.10	32.00± 3.00	7.00± 1.01	3.10± 0.22	3.53± 0.36	9.12± 0.24	2.98± 0.11	0.17± 0.02	4.54± 0.18	0.81± 0.02
	2nd season															
Canal water	0.58± 0.03	8.08- 8.13	11.12± 0.12	3.54± 0.08	4.00± 0.34	0.51± 0.06	2.20± 0.07	6.00± 0.54	3.76± .23	0.63± 0.02	0.23± 0.06	2.87± 0.05	0.14± 0.04	0.11± 0.01	1.22± 0.13	0.02± 0.01
Waste water	5.11± 0.11	7.91- 7.95	28.15± 1.65	26.34± 0.70	15.65± 0.90	12.12± 0.45	6.12± 0.89	38.09± 2.54	6.50± 0.87	3.33± 0.11	4.12± 0.51	12.32± 0-70	3.50± 0.09	0.22± 0.03	5.78± 0.11	1.03± 0.03
IWC	3.00	6.50- 9.00	10.00	3.00	20.00	5.00	6-12	5.00	1.50	0.75	0.20	5.00	0.20	0.20	2.00	0.010

 # Means of three samples ± SD.
IWC: Irrigation water criteria, US EPA 1992

RESULTS AND DISCUSSION

1. Soil characterization and contamination: Table (1) summarizes the physical and chemical characteristics of soil samples, including both wastewater-irrigated soil and canal-irrigated soil (reference soil). Soil pH was significantly affected by the wastewater irrigation. In the wastewater-irrigated soils, the electrical conductivity (EC) of soil paste extract was significantly affected by the wastewater irrigation compared to the reference soil. As in 2008, the EC values ranged from 3.25 to 3.45 dSm^{-1} in surface and sub-surface layers of wastewater-irrigated soil, while the corresponding values for reference soil were between 0.44 and 0.62 dSm^{-1}. Calcium carbonate content was significantly higher in the surface than in the sub-surface soil layers in both treatments and was significantly higher in the surface layer of the wastewater-irrigated soil compared to the reference soil (Table 1). However, the sand, silt, and clay fractions contents were not significantly different in wastewater-irrigated soil compared with reference soil in the two seasons (Table 1). The cation exchange capacity (CEC) of wastewater-irrigated soil was significantly different compared with reference soil (Table 1). Organic matter was significantly higher in the surface layer of wastewater-irrigated soil compared with the reference soil (Table 1). The available macronutrients N, P, and K contents were significantly higher in wastewater-irrigated soil compared with canal water-irrigated soil (Table 1). This increase in the macronutrients contents may be due to the presence of high concentrations of macronutrients in wastewater and organic materials. The disposal of sewage effluents to surface waters and subsequently to agricultural land has been employed to improve soil physical and chemical conditions, as a source of plant nutrients (El-Nennah *et al.*, 1982). As shown in Tables (1 & 2), irrigating with wastewater led to gradual increase in total and available forms of nutrients and heavy metals in soils of the two seasons 2008 to 2009 (Tables 1 and 2). Similar results indicated the accumulation of heavy metals in top soils irrigated with sewage effluents (El-Nennah *et al.*, 1982). Other authors have cited evidence of increases in extractable Cu, Zn, Ni, Pb, Mn and Cd following sewage application to soils (Mapanda *et al.*, 2005; Mireles *et al.*, 2004 and Khan *et al.*, 2008). In the wastewater-irrigated soil, heavy metal (Cd, Mn, Cu, Ni, Pb, and Zn) concentrations were significantly higher than the corresponding concentrations of reference soil. The results indicated that all heavy metal concentrations ,except for Mn, were above the maximum allowed levels for soils (Alloway, 1995), and there was substantial buildup of Cd, Mn, Cu, Ni, Pb and Zn in the wastewater-irrigated soil compared to the reference soil. In 2008, average PLI index of surface soil for Cd, Mn, Cu, Ni, Pb and Zn was 17.33, 17.52, 11.07, 3.65, 25.47, and 4.55, respectively, and was 9.06, 25.74, 15.05, 3.71, 31.92, and 3.41, respectively for sub-surface soil. Similar results were observed in the second season. Our results are in agreement with those reported by Mapanda *et al.* (2005) and Khan *et al.* (2008).

2. Water characteristics: As compared with the USEPA (1993) maximum allowed irrigation water criteria the data presented in Table (3), showed an increase in soluble salts, boron, and heavy metals *i.e.* Cd, Cu, Mn, Ni, Pb, and Zn in wastewater as compared to canal water. This increase can be attributed to the pollution of agricultural drainage water with untreated municipal water. The EC of wastewater was more than 3 dSm^{-1} in both sampling seasons. The concentrations of chloride, sodium, magnesium, nitrate, bicarbonate, and boron were more than the US EPA criteria. Moreover, the soluble forms of heavy metals except for Mn were more than the allowed levels in irrigation water. However, the calculated SAR and calcium content in the wastewater were less than US EPA (1993). We observed that wastewater-irrigated trees were smaller in size and had leaves with brighter green color as compared with canal water-irrigated trees, although no symptoms of chronic toxicity have appeared on wastewater-irrigated trees. The increase in EC, boron and heavy metals in wastewater may cause a gradual accumulation in soil and uptake by trees which can lead to a potential problem. On the other hand, a metal such as cadmium may not affect the growth of citrus tree, but it can render a health hazard when consumed by humans or animals (El-Nennah *et al.*, 1982 and Melia *et al.*, 2002).

3. Macro elements and heavy metals partitioning in citrus trees: Macro elements (N, P, and K) concentrations in roots, leaves and fruits of trees grown in wastewater-irrigated soil increased significantly as compared with trees grown in the reference soil (Figure 1). The N concentration was 1.7 %, 2.6%, and 1.8% in root, leaves and fruits, respectively of orange trees grown in canal water-irrigated soil, for the first season. The results of second season were close to that of the first season. However, in wastewater-irrigated soil, the N concentration was higher than the concentrations in all plant tissues in the reference soil (Figure 1). Similarly, the concentrations of P in tissues of trees grown in wastewater-irrigated soil were higher than the concentrations in tissues of trees grown in reference soil in the two growing seasons (Figure 1). The concentrations of P in the second season were 0.83, 0.74, and 0.78% in roots, leaves, and fruits of orange trees grown in wastewater irrigated soil, respectively, whereas, the concentration of P in the first season was 0.53, 0.47, and 0.55% in roots, leaves and fruits of orange (Figure 1).This can be due to the accumulation of nutrients in the soil as a result of continuous irrigation with wastewater (Khan *et al.*, 2008). Also, the results of current study indicated that the concentrations of K in different tree tissues were significantly higher in trees grown in wastewater-irrigated soil compared with reference soil (Figure 1). These results indicated that the wastewater may be a source of some nutrients such as N, P and K and contains substantial amounts of beneficial nutrients which are creating opportunities to agricultural production. These results were in agreement with the results of Chen *et al.*, (2005), Singh *et al.*(2004) and Elsokkary (2001). Heavy metals concentrations in different parts of trees grown in wastewater-irrigated soils were higher than those in trees grown in reference soil as well as from the maximum allowed daily intake (MADI) for human (Levander, 1990), as illustrated in Figures (2 &3).

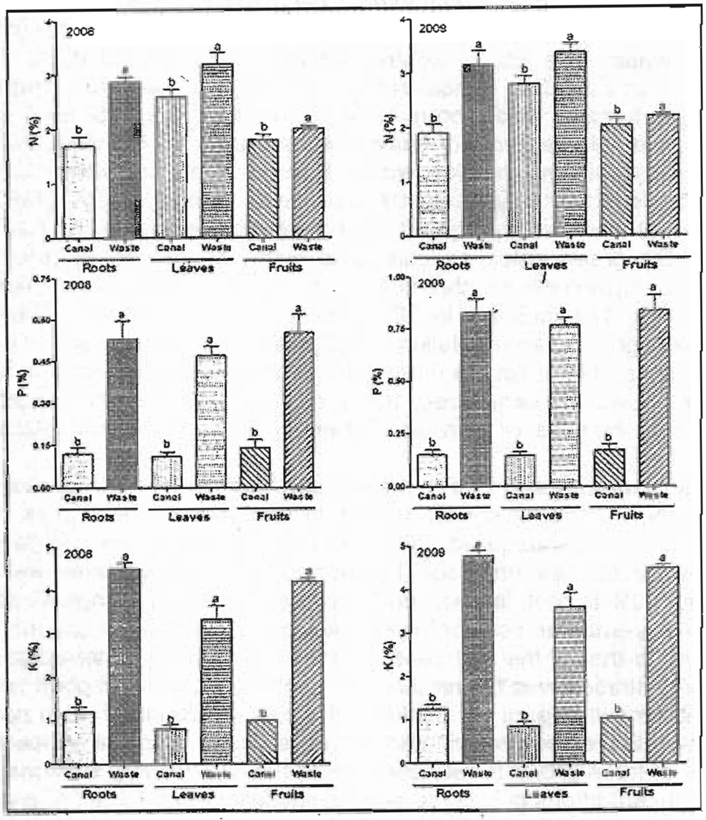

Figure (1): **Nitrogen (N), phosphorus (P), and potassium (K) levels in tissues of Valencia orange trees as influenced by irrigation with waste and canal waters. T bars = SD, n = 5. Lowercase letters above SD bars indicate significant difference by t-test.**

The Cd concentrations ranged from 5.12 ppm to 10.99 ppm in trees grown in wastewater-irrigated soil, and were significantly higher than trees grown in the reference soil (Figure 2). In all tree tissues, concentrations of Cd exceeded the MADI limit (0.07 ppm). These results were confirmed with the results of the second season. Similarly, the Ni concentrations were significantly higher in wastewater-irrigated trees and ranged from 28.19 ppm to 35.17 ppm in the first season and from 32.13 ppm to 40.40 ppm in the second season (Figure 2). In the tree plant tissues, concentrations of Ni exceeded the MADI limit (0.35 ppm). The Pb concentrations varied between 48.23 ppm and 70.18 ppm, in trees grown in wastewater-irrigated soil and were significantly higher than trees grown in the reference soil, and exceeded the MADI limit for Pb (0.25 ppm) in the two seasons (Figure 2). Also, the Cu concentrations were significantly higher in trees grown in wastewater-irrigated soil than trees grown in the reference soil, and ranged from 24.82 ppm to 28.26 ppm in the first season and from 27.98 ppm to 33.13 ppm in the second season (Figure 3). In all tree tissues, concentrations of Cu exceeded the MADI limit (1.50 ppm). Similarly, the Mn concentrations varied between 19.19 ppm and 40.14 ppm, in trees grown in wastewater-irrigated soil and were significantly higher than trees grown in the reference soil, and exceeded the MADI limit for Mn (2.65 ppm) in the two seasons (Figure 3). The Zn concentrations were significantly higher in trees grown in wastewater-irrigated soil than trees grown in the reference soil, and ranged from 162.93 ppm to 225.32 ppm in the first season and from 178.18 ppm to 259.12 ppm in the second season (Figure 3). In all tree tissues, concentrations of Zn were higher in wastewater-irrigated trees than those grown in reference soil and the concentrations exceeded the MADI limit for Zn (10.00 ppm). Results from present and previous studies (Elsokkary, 2001; Mireles *et al.*, 2004; Solis *et al.*, 2005; Liu *et al.*, 2005; Muchuweti *et al.*, 2006 and Sharma *et al.*, 2007) demonstrate that plants grown in wastewater-irrigated soils are contaminated with heavy metals.

4. Heavy metal transfer from soil to tree tissues

Typically, the soil-to-plant transfer factor is one of the key components of human exposure to metals through the food chain. In order to investigate the human health risk index (HRI) associated with wastewater-irrigated soil, it is essential to assess the PCF (Cui *et al.*, 2004). The PCF values between wastewater-irrigated and reference soils were significantly different. The mean values of PCF for heavy metals including Cu, Pb, Ni, Mn, and Cd were lower in trees grown in the wastewater-irrigated soils than trees grown in the reference soil. Mean values of PCF in wastewater-irrigated trees for Cu, Pb, Ni, Mn, and Cd ranged from 0.69 to 0.79, 0.83 to 1.21, 1.55 to 1.93, 0.65 to 1.36, and 1.68 to 3.60, respectively, in the first season, and ranged from 0.73 to 0.87, 0.84 to 1.30, 1.51 to 1.90, 0.66 to 1.41, and 1.51 to 2.98, respectively in the second season (Table 4). This was due to the high concentrations of heavy metals in the wastewater-irrigated soil as compared with the eference soil.

On the contrary, mean values of PCF for Zn was higher in trees grown in the wastewater-irrigated soil than trees grown in reference soil, and ranged from 3.00 to 4.25 in the first season and from 3.02 to 4.39 in the second season (Table 4). The trend of PCF for heavy metals in different parts of trees were in the order of Zn> Cd > Ni > Pb> Mn > Cu in wastewater-irrigated trees, and was in the order of Cd > Mn > Pb > Cu > Ni > Zn in canal water-irrigated trees (Table 4). Similarly, the PCF values for the macro elements in all tree tissues between wastewater-irrigated trees and canal water-irrigated trees were significantly different. The mean values of PCF for N, P and K were higher in trees grown in wastewater-irrigated soil than trees grown in reference soil (Table 4). The trend of PCF for macro elements in different parts of tree was in the order of P > N = K in wastewater-irrigated trees, and was P > N > K in canal water-irrigated trees (Table 4). Our results agreed with the findings of previous studies (Mapanda *et al.*, 2005; Rattan *et al.*, 2005, and Khan *et al.*, 2008).

Table (4): Macro elements and heavy metal transfer factors (on dry weight basis) for trees grown in canal and wastewater-irrigated soils.

Irrigation	Plant Concentration Factor (PCF)																	
	N						P						K					
	1st season 2008			2nd season 2009			1st season 2008			2nd season 2009			1st season 2008			2nd season 2009		
	Root	Leaf	Fruit	Root	Leaf	Fruit	Root	Leaf	Fruit	Root	Leaf	Fruit	Root	Leaf	Fruit	Root	Leaf	Fruit
Canal water	357.89	547.37	378.95	384.00	566.00	436.00	502.09	460.25	585.77	555.56	592.59	592.59	171.43	114.29	142.86	177.14	122.86	154.29
	(11.12)	(12.32)	(9.43)	(21.00)	(18.45)	(7.22)	(13.23)	(16.87)	(19.09)	(23.13)	(26.34)	(24.76)	(9.06)	(11.45)	(12.43)	(16.74)	(8.54)	(10.11)
Wastewater	509.73	566.37	355.75	513.33	575.00	381.67	1077.24	955.28	1117.89	1185.71	1057.14	1114.29	530.66	392.69	496.46	531.11	395.56	498.89
	(15.12)	(18/18)	(8.23)	(19.87)	(26.98)	(17.34)	(34.76)	(28.23)	(27.08)	(25.45)	(22.54)	(30.12)	(22.43)	(16.12)	(14.65)	(22.11)	(15.90)	(18.23)
	Cu						Pb						Ni					
Canal water	5.42	3.25	2.78	3.74	2.50	2.09	11.06	10.47	8.44	9.69	8.15	7.11	3.65	3.13	3.30	3.14	2.67	2.86
	(0.22)	(0.18)	(0.11)	(0.17)	(0.10)	(0.11)	(0.77)	(0.68)	(1.09)	(0.88)	(0.78)	(0.96)	(0.23)	(0.19)	(0.14)	(0.42)	(0.33)	(0.28)
Wastewater	0.79	0.70	0.69	0.87	0.76	0.73	1.21	0.92	0.83	1.30	0.90	0.84	1.93	1.55	1.71	1.90	1.51	1.76
	(0.09)	(0.08)	(0.06)	(0.11)	(0.12)	(0.09)	(0.15)	(0.17)	(0.19)	(0.18)	(0.22)	(0.19)	(0.28)	(0.33)	(0.46)	(0.39)	(0.36)	(0.32)
	Mn						Zn						Cd					
Canal water	12.59	9.35	4.33	4.89	3.91	2.09	3.42	2.66	1.83	3.06	2.45	1.77	8.35	7.76	6.86	12.50	10.00	16.93
	(0.99)	(1.06)	(0.88)	(0.76)	(0.72)	(0.55)	(0.87)	(0.55)	(0.33)	(0.44)	(0.18)	(0.13)	(1.06)	(0.76)	(0.77)	(0.93)	(0.88)	(1.10)
Wastewater	1.36	0.96	0.65	1.41	0.98	0.66	4.15	3.22	3.00	4.39	3.20	3.02	3.60	1.68	2.02	2.98	1.51	2.21
	(0.08)	(0.11)	(0.13)	(0.43)	(0.12)	(0.10)	(0.23)	(0.16)	(0.18)	(0.28)	(0.17)	(0.16)	(0.32)	(0.08)	(0.26)	(0.23)	(0.12)	(0.21)

Data are the average (n=5).

Numbers in parenthesis indicate the standard deviation.

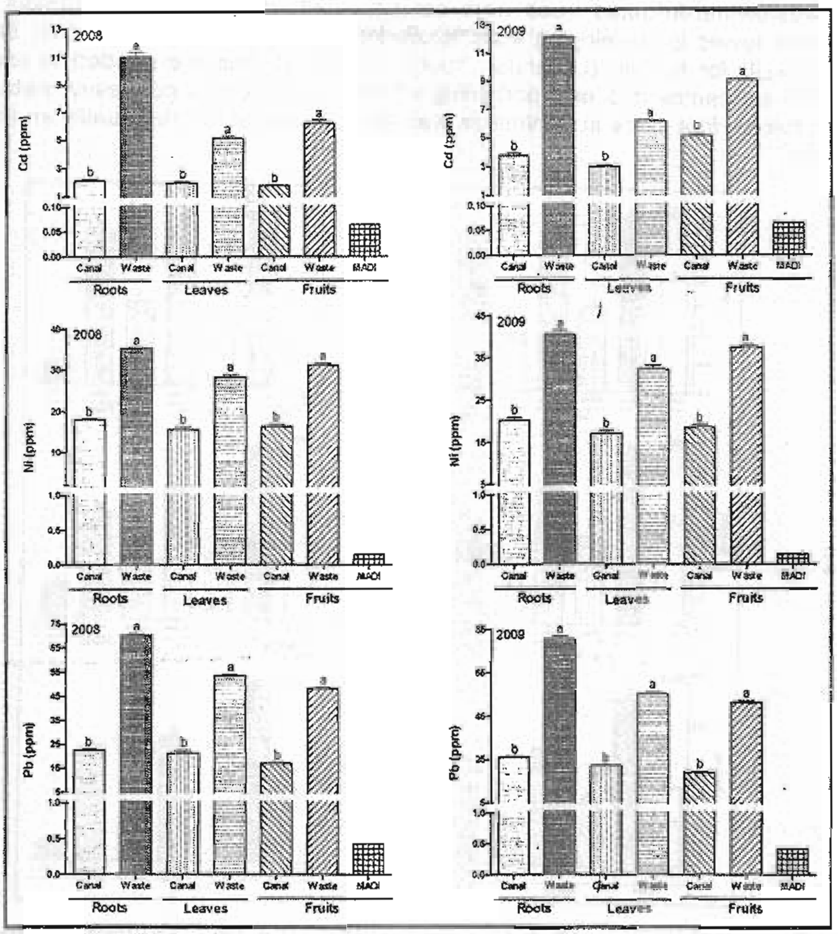

Figure (2): Cadmium (Cd), Nickel (Ni), and Lead (Pb) levels in tissues Valencia orange trees as influenced by irrigation with waste and canal waters, compared with the maximum allowed daily intake (MADI) of fruit for humans (Levander, 1990). T bars = SD, n = 5. Lowercase letters above SD bars indicate significant difference by t-test.

We concluded that application of wastewater for several years had led to changes in some soil physicochemical characteristics and uptake and partitioning of heavy metals in orange trees. Heavy metals in wastewater irrigated soils showed a substantial build-up with a significant increase over reference soil. The pollution load index values indicated that the wastewater-irrigated soil was strongly enriched with heavy metals. Furthermore, the wastewater-irrigated trees were contaminated with these heavy metals and their levels in developing fruits exceeded the maximum allowed daily intake (MADI) for human (Levander, 1990). Further studies are needed to assess the concentrations and portioning of macro elements and heavy metals in different fruit parts at harvest as well an assessment for fruit quality and shelf life.

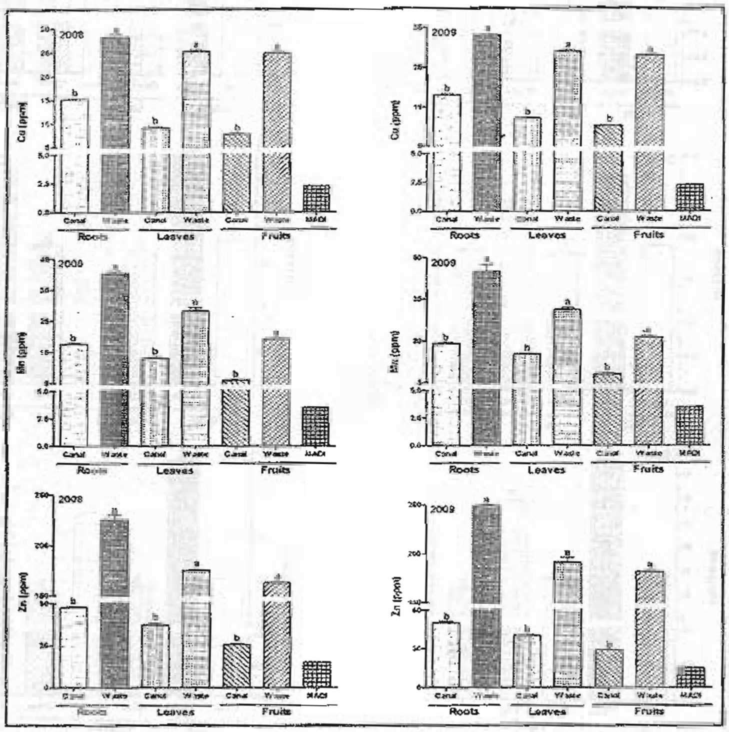

Figure (3): **Copper (Cu), Manganese (Mn), and Zinc (Zn) levels in tissues Valencia orange trees as influenced by irrigation with waste and canal waters, compared with the maximum allowed daily intake (MADI) of fruit for humans (Levander, 1990). T bars = SD, n = 5. Lowercase letters above SD bars indicate significant difference by t-test.**

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تأثيرات الري بمياه الصرف على مستوى العناصر الكبرى والعناصر الثقيلة في أشجار البرتقال فالنشيا النامية في أرض صحراوية:

١ - الإمتصاص والتوزيع أثناء مرحلة تطور الثمار

أحمد محمد مهدي*، ضياء أسامة الأنصاري** و عواد محمد حسين**
* قسم علوم الأراضي والمياه، كلية الزراعة (الشاطبي)، جامعة الإسكندرية
** قسم الفاكهة، كلية الزراعة (الشاطبي)، جامعة الإسكندرية

إن أهداف إجراء هذا البحث هي دراسة حمل التلوث في التربة وتحديد تأثيرات الري بمياه الصرف على إمتصاص وتوزيع العناصر الكبرى والمعادن الثقيلة في أشجار البرتقال الصيفي فالنشيا [*Citrus sinensis* (L.) Osbeck] أثناء مرحلة التطور النشطة للثمار في صحراء غرب النوبارية. ولقد أشارات النتائج إلى أن إستخدام ماء الصرف قد أدى إلى حدوث تغيرات في بعض الخصائص الطبيعية والكيماوية للتربة. ولقد وجد أن تركيز كل من العناصر الكبرى (النيتروجين، الفوسفور، البوتاسيوم) والعناصر الثقيلة (الكادميوم، المنجنيز، النحاس، النيكل، الرصاص، الزنك) كان أعلى في التربة التي تروى بمياه الصرف مقارنة بالتربة المرجعية التي تروى بمياه النيل العادية. ولقد إزداد تركيز كل العناصر الثقلية ماعدا المنجنيز في التربة التي تروى بمياه الصرف عن الحد الأقصى المسموح به في التربة. ولقد أشارت قيم دليل حمل التلوث إلى أن التربة التي تروى بيماه الصرف كانت محملة جدا بالعناصر الثقلية. كما أن الأشجار التي تم ريها بمياه الصرف كانت ملوثة بالعناصر الثقيلة حيث إزداد تركيزها في الثمار المتطورة عن الحد الأقصى المسموح به في التناول اليومي للإنسان. ولقد كان إتجاه معامل التركيز في النبات للعناصر الثقيلة في الجذور والأوراق والثمار للأشجار التي تروى بمياه الصرف كما يلي: الزنك > الكادميوم > النيكل > الرصاص > المنجنيز > النحاس، هذا بينما كان الإتجاه في حالة الأشجار التي تروى بمياه النيل العادية كما يلي: الكادميوم > المنجنيز > الرصاص > النحاس > النيكل > الزنك.

قام بتحكيم البحث

أ. د/ السيد محمود الحديدى	كلية الزراعة – جامعة المنصورة
أ. د/ ابراهيم حسين السكرى	خارجى