

Hazardous of Waste Water Irrigation on Quality Attributes and Contamination of Citrus Fruits

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There is an increasing concern regarding the potential health risks associated with consuming crops grown on sewage irrigated soils. The present study was carried out to assess levels of different heavy metals like iron, manganese, copper and zinc, in citrus fruits irrigated with water from two places in Jeddah; namely Al Hada site (as a control) and Al Musk Lake (as a contaminated site). Heavy metal concentrations were several fold higher in all the collected samples (water, soils and fruits) from wastewater irrigated site compared to potable water irrigated ones. Fruits collected from contaminated soils had lower firmness (30% less than that collected from the reference site), while their size, weight and their soluble solids content (SSC) increased up to 3.5 cm in diameter, 22% and 14%, respectively. We found central axis of the orange fruits irrigated with wastewater was infected by the fungus *Alternaria citri* in the form of a rot, with hardly any external symptoms. Assessment of water and soils collected from Al Musk Lake indicated that they are not suitable for any uses. Moreover, these higher levels of metal pollution in the wastewater irrigated site presented a significant threat of negative impact on human health. The results presented demonstrate that there is a risk associated with consumption of fruits and vegetables irrigated with wastewater, with the fruits still looking apparently healthy and growing well despite accumulating heavy metals to concentrations which substantially exceed maximum values considered safe for human consumption

Key words: Heavy metal, waste water irrigation, Orange, Health risk.

Access to adequate water for irrigation is a matter of increasing concern all over the globe especially in arid and semiarid areas (Al-Lahham *et al.*, 2003; Sharma *et al.*, 2007). People are using sewage for irrigation of agricultural land due to scarcity of fresh and underground water. To face the growing demand for irrigation water, nonconventional resources are often used (Palese *et al.*, 2009; Mani & Kumar, 2014).

While wastewater provides water and substantial amounts of beneficial nutrients, it leads to the potential accumulation of hazardous materials (Liu *et al.*, 2003; Chen *et al.*, 2005; Singh *et al.*, 2004). Application of industrial effluents as water source for irrigation of crop plants (Kaushik *et al.*, 2005; Rattan *et al.*, 2005) or sludge as manure are the major sources of metal contamination in agricultural land (Bose & Bhattacharyya, 2008). The threat that metals pose to the health is aggravated by their long-term persistence in the environment (Gupta & Sinha, 2007). These metals are harmful to humans and animals as they accumulate in food chain (Chandra *et al.*, 2009). Moreover, they are persistent and non-

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biodegradable and are associated with numerous serious health disorders such as diarrhea, vomiting, paralysis, convulsion, depression, and pneumonia (Duruibe *et al.*, 2007). Moreover, their effects could be carcinogenic, neurotoxic, or even mutagenic (European Union, 2002, Singh *et al.*, 2010). Moreover, contaminated water or wastewater has a great potential for transmitting a wide variety of diseases and illnesses due to presence of a wide spectrum of pathogens (Al-Nakshabandi *et al.*, 1997).

Heavy metal contamination in wastewater has been reported in a number of previous studies from developing countries (Cao & Hu, 2000; Mapanda *et al.*, 2005; Nyamangara & Mzezewa, 1999; Singh *et al.*, 2004) and wastewater irrigated soil (Cao and Hu, 2000; Mapanda *et al.*, 2005; Nan *et al.*, 2002; Nyamangara & Mzezewa, 1999; Singh *et al.*, 2004). However, there are very few empirical data from Saudi Arabia for heavy metal contamination of soil and irrigation water and its transfer to vegetable crops (Hassan & Basahi, 2013).

In peri-urban ecosystem such as Saudi Arabia, industrial or municipal wastewater is mostly used for the irrigation of crops due to its easy availability, disposal problems and scarcity of fresh water. Such practice would contribute significantly to the heavy metals content of soil (Arora *et al.*, 2008). In their a thorough study, Singh *et al.* (2010) stated that the growing problem of water scarcity has significant negative influence on economic development, human livelihoods, and environmental quality in India. Rapid urbanization and industrialization releases enormous volumes of wastewater, which is increasingly utilized as a valuable resource for irrigation in urban and peri-urban agriculture. It drives significant economic activity, supports countless livelihoods particularly those of poor farmers, and substantially changes the water quality of natural water bodies (Marshall *et al.*, 2007). Wastewater may contain various heavy metals including Zn, Cu, Pb, Mn, Ni, Cr, Cd, depending upon the type of activities it is associated with. Continuous irrigation of agricultural land with sewage and industrial wastewater may cause heavy metal accumulation in the soil and vegetables (Singh *et al.*, 2004; Sharma *et al.*, 2007; Marshall *et al.*, 2007, Qishlaqi *et al.*, 2008, Du *et al.*, 2014).

Large urban populations along with industrial activities in Jeddah generate a great volume of wastewater which is discharge in an area, called Al Musk Lake due to its bad smell. However, it is very rich in nutrients due to accumulation of wastes. People used its soil for rapid cultivation of many crops and vegetables despite the health and environmental risks associated with such practice.

The responses of citrus rootstocks to several environmental stresses other than heavy metal have been extensively studied (Arbona *et al.*, 2003; Gomez-Cadenas, 1996); however, scarce information can be found on plants ability to cope with the heavy metal stress (Podaza *et al.*, 2012), and even there is a paucity on information about the status in Saudi Arabia.

Our objective was to study the effect of irrigation by wastewater, as compared to normal potable water, on the quality attributes and contamination of citrus fruits as well as heavy metals (Na, Zn, K, Cu, Ni, Cd, Fe, Cr and Pb) and to determine their potential detrimental effects.

MATERIALS AND METHODS

Description of Sites

Wastewater accumulates in underground cesspools later is transported by truck tankers to the sewage lake for the past 10 years (Fig. 1). The lake lies in east of Jeddah (Fig 2 A) at about 130m above sea level. It contains 9.5 million cubic meters sewage water spread over an area of 2.88 km² (Fig 2 B) (El Feki *et al.*, 2011). Samples of orange fruits (*Citrus sp.*) were collected from two different sites in Jeddah, namely Al Musk Lake (irrigated with wastewater), and agricultural fields at Al Hada (Fig. 2, indicated by green star) irrigated with fresh potable water.

Sample preparation

All the collected samples were washed with double distilled water to remove airborne pollutants. The fruits were counted and weighed and air-dried for a 24 h, to reduce water content. All the samples were then oven-dried in a hot air oven at 70–80 °C for 24 h, to remove all moisture. Dried samples were powdered using a pestle and mortar and sieved through muslin cloth.

Water and soil sampling

Soil samples were collected from both locations at four positions under citrus trees. Then

were immediately put in plastic bags, tightly closed and immediately transported to the laboratory.

Water samples were collected in 500 mL plastic containers at each location. The containers were tightly closed, placed in an ice box (5°C) and then transported to the laboratory where pH, EC and total dissolved salts (TDS) were immediately determined (Al- Lahham *et al.*, 2003). The water samples were then filtered and refrigerated before metal analysis, as described earlier (Hassan & Basahi 2013).

Weight and weight loss

Fresh weight for individual fruit was taken directly upon arrival at the laboratory, and subsequently every 2 days for a period of 10 days whilst held at room temperature.

Soluble solids content (SSC)

Fruit soluble solids content was determined using a refractometer (Abbe-Refractometer No. 301, Japan, BRMER LABORHANDEL GMBH), reading SSC and converting to percent.

Firmness and diameter of fruits

Tomato fruit firmness was measured by using fruit pressure tester (EFFEGI-480011 ALFONSINE, Italy). Fruit size was determined by measuring the diameter at the maximum circumference of the fruit using a caliper.

Digestion of the vegetable samples

For metal analysis, only the edible parts of fruits were used. Three powdered samples from each source of irrigation (0.5 g each) were accurately weighed and placed in crucibles, three replicates for each sample. The ash was digested with perchloric acid and nitric acid (1:4) solution. The samples were left to cool and contents were filtered through Whatman filter paper No. 42.

Analysis of heavy metals

The elemental analysis was performed

by inductively coupled plasma optical emission spectrometry (ICP-OES) using IRIS Intrepid II XSP instrument. A sixpoint calibration procedure was applied with multi-element calibration solution (Merck ICP multi-element standard solution IV) (Hassan & Basahi, 2013).

Statistical analysis

The data of heavy metal concentrations in the plants at both sites were subjected to one-way analysis of variance (ANOVA) test, using STATGRAPHICS (Package 4) for assessing the significance of differences in heavy metal concentrations due to different irrigation practices. Least Significant Difference (LSD) was used to test the significance of differences between the concentrations of heavy metals in soil at wastewater (WW) and potable water irrigated (PW) sites. Moreover, the relationships between elemental concentrations in the fruits and the concentrations of heavy metals in wastewater and soil were assessed using correlation analysis.

RESULTS AND DISCUSSION

The physical and chemical properties of soil and water collected from both sites are given in Table 1. WW and contaminated soils (collected from Musk site) showed increases in pH by 13 and 20% compared with PW and reference soil, respectively. EC also increased by 19 and 17% in both samples, respectively. Total dissolved solids (TDS) were increased by 1- and 4-fold in WW and contaminated soils, respectively (Table 1).

The increase in EC values of soil irrigated with WW could be attributed to higher accumulation of soluble salts (Khurana & Singh, 2012). This is supported by higher pH which is due to alkalization effect of basic cations (e.g. K)

Table 1. Physical and chemical properties of water and soil collected from both experimental sites (n=10 ± SD)

Parameter	Water		LSD	Soil		LSD
	WW	PW		Al Musk	Al Hada	
pH	8.25± 0.007	7.32±0.002	0.04**	8.93 ± 0.006	7.41 ± 0.017	0.03**
EC (mS cm ⁻¹)	0.37 ± 0.02	0.31 ± 0.00	0.02**	0.41 ± 0.02	0.35 ± 0.02	0.02**
TDS (mg l ⁻¹)	786.41±56.57	341.25± 22.76	55.38***	1016±100.34	298.11±23.93	76.98***

WW= wastewater; PW = Potable water.

Different letters means that means are significantly different from each other at 0.0001 < P < 0.05), * = level of significance at 0.01 < P < 0.05; ** = level of significance at 0.001 < P < 0.01; *** = level of significance at P < 0.0001

contained in wastewater (Qishlaqi *et al.*, 2008). Such an increase in alkalinity indicates significant effect of WW irrigation (Chen *et al.*, 2009).

Levels of heavy metals in wastewater and wastewater-irrigated soils collected from Al Musk Lake were much higher than that recorded

Table 2. Concentrations of heavy metals in water ($\mu\text{g ml}^{-1}$) and soils ($\mu\text{g g}^{-1}$) collected from different sites

Parameter	Water		LSD	Soil		LSD
	WW	PW		Al Musk	Al Hada	
Pb	0.093 ^b ±0.001	0.011 ^a ±0.001	0.056*	26.37 ^b ±3.18	7.81 ^a ±2.72	4.17**
Cu	0.102 ^b ±0.021	0.051 ^a ±0.004	0.048*	145.81 ^b ±11.79	17.54 ^a ±3.02	12.83**
Cd	0.036 ^b ±0.002 ^Δ	0.014 ^a ±0.003	0.014**	6.14 ^b ±1.12 ^Δ	1.91 ^a ±0.15	2.71**
Zn	0.191 ^b ±0.011 ^Δ	0.021 ^a ±0.001	0.026*	371.24 ^b ±25.9 ^Δ	45.87 ^a ±6.28	23.85***
Ni	0.112 ^b ±0.015 ^Δ	0.045 ^a ±0.006	0.053**	76.12 ^b ±11.91 ^Δ	18.21 ^a ±3.27	11.03***
Cr	0.104 ^b ±0.001	0.046 ^a ±0.007	0.037*	80.61 ^b ±18.67	11.07 ^a ±2.19	22.73**
Fe	2.362 ^b ±0.131 ^Δ	0.532 ^a ±0.006	0.817*	20.99 ^b ±1.98 ^Δ	0.278 ^a ±0.12	6.39***
Na	9.12 ^b ±0.247 ^Δ	0.402 ^a ±0.007	1.02***	12.08 ^b ±1.45 ^Δ	0.235 ^a ±.027	4.06**
K	10.23 ^b ±1.058 ^Δ	0.169 ^a ±0.004	1.25***	14.21 ^b ±2.13 ^Δ	0.094 ^a ±.002	4.74**

Legends as table 1. Δ = levels are above safe limits. (n = 10 ± 1 SD)

Table 3. Concentrations of heavy metals in Orange fruits ($\mu\text{g g}^{-1}$ fresh weight) collected from different sites (Legends as Table 1)

Metal	Site		LSD
	Al Musk (Contaminated site)	Al Hada (Reference site)	
Pb	0.193 ^b ±0.001	0.037 ^a ±0.001	0.051**
Cu	0.156 ^b ±0.009	0.056 ^a ±0.004	0.027**
Cd	0.104 ^b ±0.008	0.034 ^a ±0.003	0.011**
Zn	0.321 ^b ±0.021	0.032 ^a ±0.001	0.025**
Ni	0.112 ^b ±0.011	0.061 ^a ±0.006	0.018**
Cr	0.104 ^b ±0.001	0.007 ^a ±0.007	0.027**
Fe	4.044 ^b ±0.531	0.241 ^a ±0.006	0.537***
Na	7.652 ^b ±0.477	0.362 ^a ±0.007	1.078***
K	8.468 ^b ±0.958	0.210 ^a ±0.004	2.055***

Table 4. Relationships between concentrations of heavy metals in orange fruits and their concentrations in WW and soil (r: correlation coefficient; ***p<0.001). n= 15

Element	r	
	WW	Soil
Pb	0.219***	0.353***
Cu	0.754***	0.672***
Cd	0.346***	0.328***
Zn	0.834***	0.673***
Ni	0.674**	0.569***
Cr	0.419***	0.528***
Fe	0.645***	0.710***
Na	0.852***	0.719**
K	0.934***	0.875***

in potable water or soils collected from Al Hada experimental station (Table 2). Concentrations of heavy metals ranged from 0.011 $\mu\text{g ml}^{-1}$ in water collected from a potable for Pb to 9.12 $\mu\text{g ml}^{-1}$ in water collected from Wastewater in Na. Moreover concentrations of Cd, Zn, Ni, Fe, Na and K were above safe limits in both water and soil samples collected from sites receiving wastewater (WHO, 1993; European Union, 2002; 2006). Chary *et al.* (2008) highlighted the potential risk of heavy metals from consuming food grown on sewage irrigated soils.

Heavy metals are non biodegradable, and irrigation with wastewater causes cumulative increases in the soils (Rattan *et al.*, 2005, Du *et*

al., 2014). Our results indicated that irrigation with wastewater is the main source heavy metals contamination in the soil of the study area. The ANOVA analysis (Table 2) showed that the concentrations of individual heavy metals in wastewater-irrigated soils (Al Musk Lake) were significantly higher ($P < 0.001$) than the reference soils (Al Hada site), indicating that the application of wastewater has seemingly enriched the soils in heavy metals. There is a huge body in literature indicated that the use of untreated wastewater causes increase in pH, EC, build up of heavy metals in topsoil above Maximum Permissible Limits and their excessive accumulation in vegetables and fruits due to their continual addition of through long-term wastewater application (Liu *et al.*, 2005; Lucho-Contantino *et al.*, 2005; Muchuweti *et al.*, 2006; Qishlaq *et al.*, 2008; Khurana & Singh, 2012).

High levels of heavy metals in the soils of Al Musk Lake pointing adverse impacts, and this in agreement with the results of (Chary *et al.*, 2008). However, to the best of our knowledge, this is the first study conducted in KSA on the metal contamination of soils resulting from disposal of wastewater at the study area and it shows a clear impact on environmental quality that would pose

a threat to agriculture and health.

Table 3 shows that high levels of potentially hazardous metals are frequently found in fruits grown at Al Musk area. Some metals increased by 2-fold (e.g. Cu and Cd), others increased by 4- and 9-fold (Pb and Zn, respectively), while Cr, Fe, NA and K were increased greatly by 16-, 20- and 39-fold, respectively (Table3).

Table 4 shows a least-squares linear regression analysis between the concentrations of the metals in orange fruits and in that in WW and soils (Table 4). There was a strong correlation ($p < 0.001$) between the concentration of heavy metal in fruits and that in wastewater and soil. This in agreement with the general view in the literature, that the metal contents observed in the crops depend on the physical and chemical nature of the soil which in turn controls heavy metal bioavailability (e.g. Sharma *et al.*, 2007; Khurana & Singh, 2012). However, our results contradict the results of Qishlaqi *et al.* (2008), who reported that the occurrences of higher concentration of heavy metals in plants may be due to factors other than soil concentration.

Irrigation of fruits increased yield parameters (weight and number of fruits) in the present study by about 22% (Table5). Such

Table 5. Yield and quality parameters of fruits irrigated (Legends as Table 1; ns = Not Significant)

	WW (Al Musk)	PW (Al Hada site)	LSD at $P \leq 0.05$
Yield Parameters			
FW of fruit (gm)	174.29 ^b ± 22.65	142.68 ^a ± 11.54	17.67***
No of Fruits/tree	725.92 ^b ± 45.78	596.46 ^a ± 46.36	179.59***
FW of fruits/tree (Kg)	124.27 ^b ± 14.76	101.45 ^a ± 15.25	18.96**
Quality parameters (n=100 ± SD)			
Diameter of fruit (cm)	13.34 ^b ± 2.26	9.94 ^a ± 1.57	1.98**
Firmness (Kg)	2.97 ^a ± 0.19	4.24 ^b ± 0.28	1.52***
SSC (%)	6.47 ^b ± 0.52	5.54 ^a ± 0.61	0.94*
pH	4.36 ± 0.36	4.21 ± 0.28	0.45 (ns)

Table 6. The weight loss (%) of harvested orange fruit irrigated with either wastewater or a potable water

Treatment	Wight (gm)					Wight loss (%)
	W1	W2	W3	W4	W5	
WW	174.29	154.79	133.25	113.9	NA	35
PW	142.68	130.12	121.34	117.26	110.67	22
LSD	17.67***	14.93***	11.25***	11.34***	NA	

increases in number and fresh weight of fruits were reflected in an increase in the size of fruits, as their diameters were increased by 34% (Fig. 2 C). These results are expected as waste water contains nutrients and dissolved organic matter that enriches soils and absorbed by plants.

On the other hand WW usage is often associated with significant health risks because of the presence of high concentrations of human pathogens, enteric in origin (Shahalam *et al.*, 1998; Yan *et al.*, 2008; Palese *et al.*, 2009; Singh *et al.*, 2010; Khurana & Singh, 2012). This assumption was supported by our finding that the fruits that were irrigated with WW were less firm (about

50% lower than those irrigated with PW) (Tab 5). This lower firmness could be attributed to the fungal infection with *alternaria* that appeared in the central axis of the orange in the form of rot and works its way outward with hardly any external symptoms (Fig. 3 D). SSC and diameter of fruits irrigated with WW were increased by 17 and 34%, respectively, while pH showed insignificant increase (0.15 point) (Table 5). The reduction in pH is very comparable to the reduction reported in Jordan by Shahalam *et al.* (1998), as they reported a 0.2 unit in the case of freshwater irrigation. It indicated a tendency of the wastewater to lower the soil pH slightly.



Fig. 1. Turks disposing waste water in the lake

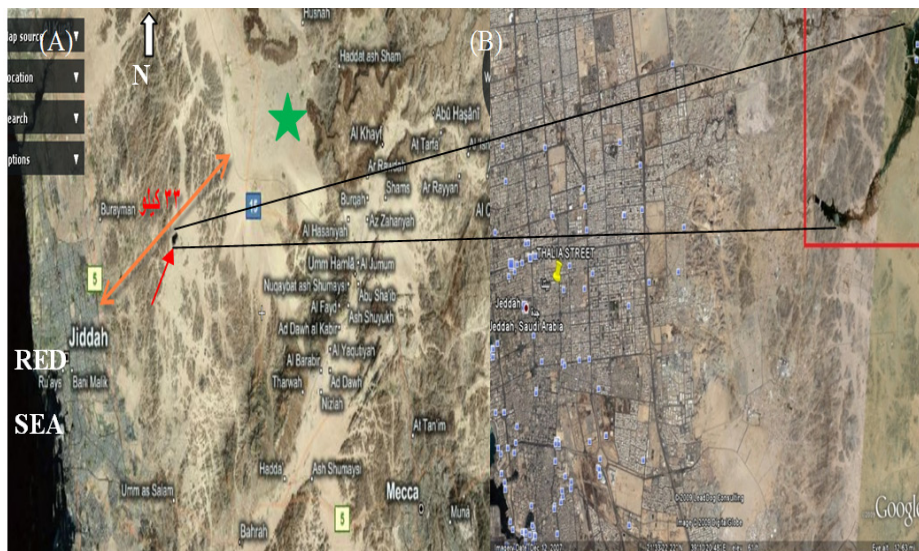


Fig. 2. (A) A map of Jeddah showing the collection sites; Al Musk Lake (where plants are cultivated in contaminated soils and irrigated with wastewater) indicated by Red arrow and a control site “Al Hada” (where plants are irrigated with a potable water) indicated by a green star. (B) shows the shape of Al Musk Lake on the map of Jeddah

Fruits irrigated with WW lost 35% of their water content within 8 days and fruits were totally rotten afterward, while those irrigated with PW lost only 22% and remained intact till end of measurements (Table 6).

Wastewater irrigation was widely used in the past decades, in the countries lacking water resources (Gao and Li, 1991; Zhou and Gao, 1995; Mapanda *et al.*, 2005; Chary *et al.*, 2008). However, wastewater often contains considerable amounts of contaminants, such as heavy metals, organic toxicants, and human pathogens, which

can induce potential risks to human health through accumulation and persistence in soil, (e.g. Yan *et al.*, 2008). The bad and unpleasant odor of WW and soils indicate presence of different types of pathogens which could further become potential source of entry into human through food chain, people who consume the crops, causing health problems to farm workers, and in the people who inhale aerosols generated from the applications of the wastewater (Chary *et al.*, 2006; Zhao *et al.*, 2014).



Fig. 3. Orange fruits collected from a controlled area irrigated with potable water (a) with health fruits (b). Fruits from a contaminated site irrigated with wastewater (c) have unhealthy fruits, showing rotting starting in the central axis of the orange and works its way outward (d)

CONCLUSIONS

The present study highlights that there is a substantial build-up of heavy metals in orange irrigated with wastewater and people consuming vegetables grown in wastewater-irrigated soils ingest significant amount of these metals. Such accumulation of heavy metals in food stuff causing potential health risks to consumer. Regular monitoring of levels of pathogens and heavy metals from effluents and sewage, in vegetables and in other food materials is essential to prevent excessive build-up of these metals in the food chain.

Saudi authority (Presidency of Meteorology & Environment “PME”) should

enforce a proper strategy for disposal of domestic and industrial wastes. Moreover, heavy fines should be charged for any organizations found dumping wastes or cultivating on wastes.

Awareness programs should be launched nationwide on the dangers of illegal disposal of wastes to life and the food chain, as well as dangers of consuming food of unknown source. We strongly recommend not consuming any food and vegetables from urban roadsides

In conclusion, our work gains significance indicating the need for proper disposal of sewage and further abatement of metal pollution and associated risk due to the consumption of foods grown on sewage wastewaters.

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