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Long-term Effect of Mixed Wastewater Irrigation on Soil Properties, Fruit Quality and Heavy Metal Contamination of Citrus

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Abstract Wastewater contains essential elements for plant growth as well as heavy metals which may be toxic for plant and human. In the northern regions of the Nile Delta, mixed water (wastewater mixed with fresh water) is used in land irrigation long time ago. The present study was conducted to assess the impact of long-term irrigation with mixed water (MW); compared to Nile fresh water (FW), on "Navel" orange [Citrus sinensis (L.) Osbeck] orchards (aged 10 to 12 years). Some soil properties, yield, fruit quality and heavy metals contamination were evaluated in three orchards irrigated with mixed water and compared with another three orchards irrigated with Nile water. The Results indicated that long term application of MW induced significant ($p<0.05$) increase of soil pH and Ec, particularly in the surface layer. In comparison with FW irrigated soil, mixed water resulted in accumulation of K, Fe, Mn, Cu, Zn, Ni and Na in the soil. On the other hand, MW irrigation increased yield parameters (fruit weight and yield/tree), soluble solids content and Brix/acid ratio. Fruit is still looking apparently healthy despite increasing of K, Fe, Mn, Zn, Ni and Na in its peel and pulp. It can be concluded that MW irrigation may be useful in the reduction of fertilizers applied to orchards. Whereas, there is a potential risks associated with crops consumption that irrigated for a long-term with mixed water. Therefore, we have to be careful about mixed water irrigation and use it under controlled conditions to protect soil properties and minimize hazards from heavy metal accumulation in agricultural products and the food chain.

Keywords: *mixed water, irrigation, heavy metals, soil, peel, pulp, orange*

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1. Introduction

Access to sufficient water for irrigation is a matter of increasing concern all over the world, especially in arid and semiarid regions. In Egypt, the total annual water supply from the River Nile, rainfall along the Mediterranean coast and deep groundwater is approximately 57.7 billion m^3. However, the yearly water requirements reached around 72.4 billion m^3 [1]. This negative water balance becomes serious with time. The northern regions of the Egyptian Nile Delta suffer from the shortage of water used for land irrigation. Farmers of this regions are obligated to reuse the wastewater from the surrounding drainages in irrigation [2,3,4]. Official and non-official pumps are used to push wastewater from drainage ditches and direct it back into the irrigation canals to produce what farmers called "Mixed water". Drainage water is different in quality to irrigation water so as the two waters are mixed together the quality of water shifts [5].

Mixed water irrigation could be an unconventional way to avoid water shortage. In Egypt, mixed water increases the available water for agricultural use by 20% [5]. It is a cheap source that does not require much infrastructure; just pumps to push the drainage water back into the irrigation system [5,6]. Mixed wastewater supplies a part of essential nutrients for plant productive and soil fertility. On the other hand, long-term irrigation with industrial, municipal and sewage wastewater may restrict soil function and nutrients uptake. In addition, there is an increasing concern about the impact of the water quality on the crop itself and on the human as the end user of the crops [7].

Heavy metals contamination in soil and wastewater irrigated crops have been reported in a number of previous studies in Egypt and other developing countries [5,6,8,9,10]. Wastewater irrigation considered as the major source of metal contamination in irrigated soils. Thus, it may contain various heavy metals such as Fe, Hg, Cd, Pb, Cu, Cr, Zn, Ni and Mn; depending up on the type of activities it is associated with [11,12]. Some of these metals transferred to food chain which can affect food quality and cause serious health hazarded to human being and animals [7,9,10]. That may pose potential environmental and health risks in the long term [13].

In Egypt, mixed water is used to irrigate many important crops consumed in local markets and exporting. Citrus is one of the most important fruit crops in the world. In 2012, the production of Egyptian citrus reached 3,980,151 tonnes [14]. Citrus peel and pulp are used in many different ways. Fruit pulp can be used fresh, juice or caned as it is a very important source of vitamin C. The citrus peels are rich in nutrients and contain many phytochemicals such as tannins, saponins, etc. [15] and they can be efficiently used as drugs or food supplements. The effect of wastewater and reclaimed water irrigation on woody trees, vegetables and crops rather than fruit crops have been extensively studied [13,16,17]. However, scarce information can be found on the response of crops to mixed water irrigation and its effect on heavy metal contamination in different parts of the fruit.

Therefore, the present study was conducted in the northern regions of Nile Delta, Egypt, to evaluate the long-term (at least 10 years) effect of mixed wastewater irrigation; compared to fresh Nile water, on soil properties, yield and fruit quality of Navel orange [Citrus sinensis (L.) Osbeck], and also to assess the accumulation level of some heavy metals in the irrigated soil, and fruit peel and pulp.

2. Materials and Methods

2.1. Site Description

The present study was conducted in 2012 and 2013 seasons, at Kafr El-Sheikh governorate, north of Nile Delta, Egypt. The study confined to six commercial Navel oranges orchards cultivated in two sites namely; Baltim and Kafr El-Sheikh city (Figure 1). Orchards in the first site were irrigated, since planting, with mixed water produced from the official pumping station No.5 which mixes wastewater from El-Gharbyia main drain with the River Nile fresh water. El-Gharbyia main drain is an open drain runs in the Nile Delta from the south to the north near El-Burlulls Lake. This drain receives wastewater from the factories in addition to agricultural and sewage water from the surrounding areas [18]. The second site (Kafr El-Sheikh city) irrigated with Nile fresh water was used for comparison.

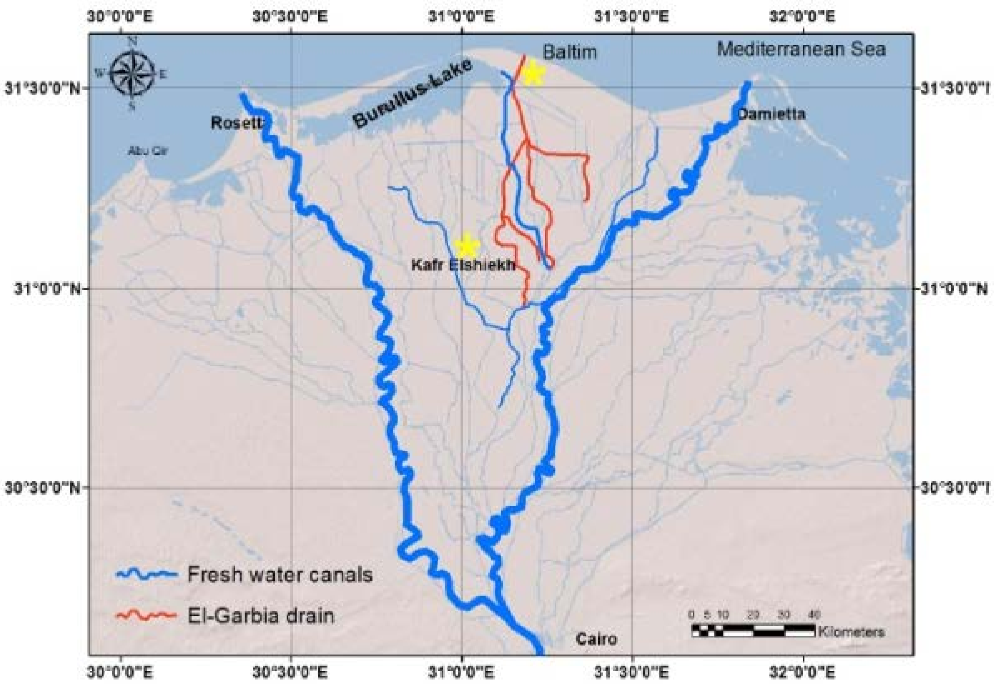

Figure 1. Map of the studied region

Three orchards with trees in age of 10 to 12 years were selected in each site. The surface irrigation is applied based on tree water-use, which varied seasonally in response to the climate. The chosen orchards apply chemical fertilization and other agricultural practices as recommended by Egyptian Ministry of Agriculture and Land Reclamation [19].

2.2. Water Analysis

Five water samples were collected from the main irrigation canals of each site in 500 ml chemically resistant plastic bottles, filtered, preserved with concentrated nitric acid and stored at 4°C. Electric conductivity (Ec), total dissolved solids (TDS) and pH of water samples were determined directly in the irrigation canals using pH-TDS meter "CRISON-MM40". Concentrations of elements and heavy metals in water samples; including potassium (K), iron (Fe), manganese (Mn), copper (Cu), zinc (Zn), cadmium (Cd) and nickel (Ni), were determined using Atomic Absorption "Perkin-Elmer, Model AAnalyst 100"), while concentration of sodium (Na) was measured using Flame Photometer "air-acetylene burner" according to [20].

2.3. Soil analysis

Soil samples were collected from nine points in each orchard at a depth of 0-30 and 30-60 cm. Samples were air-dried, crushed and passed through a 2 mm-sieve. Ec and pH of soil samples were measured in the lab according to [21]. Then, samples from each orchard were mixed together; 1gm of soil was digested and analyzed for elements and heavy metals concentration as described before in water analysis.

2.4. Fruit Yield and Quality

At harvest time, fruit yield was recorded as yield /tree for five trees in each orchard. Thirty mature fruits were randomly collected from each orchard to evaluate fruit quality. Fruit weight as well as soluble solid content (Brix), titratable acidity (TA), Brix/TA ratio, total ascorbic acid (V.C) and pH in fruit juice were measured according to [22].

To determine element concentrations in fruit peel and pulp, Fruits samples were washed and cleaned by distilled water. Twenty grams of peel as well as thirty grams of pulp (four replicates per orchard) were digested with sulphuric, nitric and perchloric acid as described by [23]. The concentrations of K, Fe, Mn, Cu, Zn, Cd, Ni and Na in digested samples were analyzed as described previously. The concentration of each element was expressed as mg.100 g^{-1} fresh weight.

2.5. Statistical Analysis

Data were statistically analyzed according to [24]. Mean values of water and soil properties, in mixed and fresh water irrigated sites were compared using t-test. Fruit quality was analyzed as completely randomized design using analysis of variance (ANOVA) test. All statistical analysis was carried out using the SPSS Inc. computer program, at the 0.05 level of significance.

3. Results and Discussion

3.1. Physico-chemical Properties of Mixed and Fresh Water

The mean values of physico-chemical properties of collected water samples are presented in Table 1. Data revealed that MW has higher pH, Ec and TDS by 1, 3 and 13- fold, respectively. These values indicated the saline nature of mixed water. Although, high TDS (over 2000 mg. L^{-1}) of irrigation water causes severe problems [25], farmers in Baltim used this water (8700 mg.$L^{-1)}$ in crops irrigation many years ago.

Table 1. Physico-chemical Analysis of Water Samples

Water properties	Unit	Mixed water	Fresh water	Standard limit for irrigation^{y}
Ec	ds. m^{-1}	1.36^{a}	0.42^{b}	<3.0
TDS	mg. L^{-1}	8700^{a}	680^{b}	<2000
pH	…	8.2^{a}	7.4^{b}	6.5-8.4
K	mg. L^{-1}	27.01^{a}	5.38^{b}	0.2
Fe	mg. L^{-1}	n.d.^{y}	n.d.	5.0
Mn	mg. L^{-1}	6.10^{a}	1.40^{b}	0.2
Cu	mg. L^{-1}	0.43^{a}	0.57^{a}	0.2
Zn	mg. L^{-1}	0.73^{a}	0.68^{a}	2.0
Cd	mg. L^{-1}	n.d.	n.d.	0.01
Ni	mg. L^{-1}	0.55^{a}	0.18^{b}	0.2
Na	mg. L^{-1}	914.5^{a}	40.2^{b}	184

Different letters that followed mean values indicated significant differences at $p<0.05$

^{x} n.d.: Not detected

^{y} Wastewater quality recommended for agricultural use [27,28,29].

The concentrations of K, Mn, Zn and Ni in MW were two times or more than in FW, particularly for potassium which was 5 times greater. These results were noticed by [26] who reported higher values of heavy metals in El-Rahawy drain than those in the River Nile water. Iron and cadmium were not detected neither in mixed nor in fresh water. On the other hand, Mn and Cu concentration in both MW and FW exceeded the maximum recommended concentrations according to FAO wastewater quality guidelines for agricultural use. Fresh water contamination may be attributed to the addition of Cu and Mn as micro nutrients in the surrounding orchards.

Sodium is the most troublesome major element in the irrigation water. Mixed water had higher sodium (914.5 mg.L^{-1}) than recommended concentration (184 mg.L^{-1}) for agriculture use [27]. This excessive concentration of sodium can cause soil physical problem and reduce water uptake by plant roots [25].

3.2. Effect of Mixed Water on the Irrigated Soil

3.2.1. Soil pH and Ec

Soil pH is an important factor that affects nutrient availability and absorption by plant roots. Soil Ec and pH had increased significantly by using MW for a long time as compared to FW irrigated soil (Fig. 2). This is probably due to the alkaline nature and high concentrations of chemical substances in MW (Table 1). The same results were obtained by [7,11,30] who reported that application of wastewater increased soil pH due to the alkalization effect of basic cations such as Na, Ca and Mg in the water. The pH values were significantly ($p<0.05$) lower in the topsoil layer than in subsoil, while soil Ec was higher in the topsoil layer, in both sites.

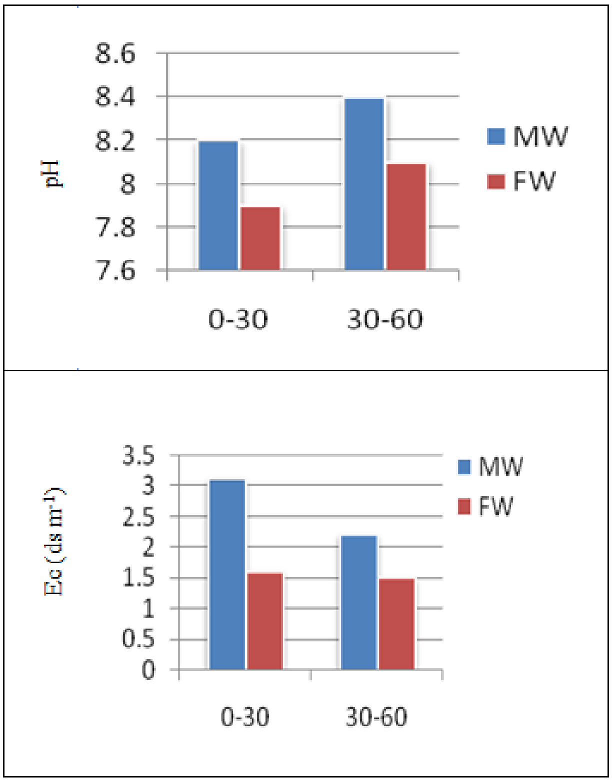

Figure 2. Mean values of pH and Ec of soils irrigated with mixed (MW) and fresh water (FW) under two depths

3.2.2. Metals Contamination in Soil

The ANOVA analysis (Table 2) showed that the level of metals in MW irrigated soils were significantly higher ($p<0.05$) than the reference soils (Kafr El-Sheikh city) these results indicated that the application of mixed water had seemingly enriched the soils in metals. My findings are in harmony with [31] who reported that heavy metals contamination in agricultural environments attributed to atmospheric fall-out, pesticide formulations and contamination by chemical fertilizers, in addition to irrigation with water of poor quality that contaminated with trace elements.

Moreover, Data in Table 2 demonstrated that all elements concentration were higher in soil than water. Although the heavy metals concentration was relatively low in the irrigated wastewater; its continuous application for many years could lead to accumulation of heavy metals in the soil [12]. The average concentration of Cu, Zn, Cd and Ni in the soil irrigated with MW is higher than the acceptable level for Germany maximum limited concentration [32] that could result in toxicity of plants and affecting food quality and safety. Elements such as; K, Fe, Mn, Cu and Zn, are necessary for plant growth as macro or micro nutrients, so we must reduce fertilizer applications when we use mixed water irrigation to protect soil and crops from trace elements accumulation.

Table 2. Mean values of element concentrations in soils irrigated with mixed and fresh water

Element	Unit	Mixed water	Fresh water
K	$mg.kg^{-1}$	126^{a}	51.0^{b}
Fe	$mg.kg^{-1}$	4107^{a}	1054^{b}
Mn	$mg.kg^{-1}$	452^{a}	81.7^{b}
Cu	$mg.kg^{-1}$	34.2^{a}	nd^{b}
Zn	$mg.kg^{-1}$	77.6^{a}	17.2^{b}
Cd	$mg.kg^{-1}$	0.687^{a}	0.703^{a}
Ni	$mg.kg^{-1}$	80.1^{a}	66.8^{b}
Na	$mg.kg^{-1}$	5221^{a}	2255^{b}

Different letters that followed mean values indicated significant differences at $p<0.05$.

Mixed water irrigation increased sodium concentrations by 23 and 2.5 times in water and soil samples, respectively compared with those of FW. The decrease of sodium level in the soil compared to its level in the water may be due to leaching properties of sandy soil, so sodium is more readily leached from the soil profile. Other researchers reported similar increases in the salt content in the soil after wastewater irrigation [33].

3.3. Effect of Mixed Water Irrigation on Citrus

3.3.1. Yield and Fruit Quality

Fruits collected from the two sites were looking apparently healthy. The effect of MW irrigation on yield parameters of Navel orange was reported in Table 3. Mixed water irrigation increased yield parameters by about 15 and 18% for fruit weight and about 25 and 29% for fruit yield/tree in the first and second season, respectively. These results are expected as mixed water contains nutrients and dissolved organic matter that enriched soil and absorbed by plants [7].

Fruit weight in the 2^{nd} season and yield/tree in both seasons were significantly differed among orchards in the same site. In addition, the interaction between water source and orchards was significant in both seasons. These results proved that there were other agricultural applications that had affect on fruit weight and yield in addition to the source of irrigation water.

Table 3. Yield parameters affected by two irrigation sources in 2012 and 2013 seasons

Water source		Fruit weight (g)		Yield/tree (kg)	
	Orchards	2012	2013	2012	2013
Mixed water	1^{st}	240	227	35	31
	2^{nd}	242	275	32	36
	3^{rd}	235	259	38	37
	Mean	**239**	**254**	**32**	**34.7**
Fresh water	4^{th}	211	202	22	29
	5^{th}	204	220	30	25
	6^{th}	209	223	25	27
	Mean	**208**	**215**	**25.7**	**27**
Significance^{z}					
Water source		*	*	*	*
Orchard		NS	*	*	*
Water source* orchard		*	*	NS	NS

Table 4. Navel orange fruit quality affected by two irrigation sources in 2012 and 2013 seasons

Water source		SSC (Brix)		Titratable acidity (%)		Brix/acid Ratio		Juice pH		Ascorbic acid ($mg.100\ g^{-1}$)	
		2012	2013	2012	2013	2012	2013	2012	2013	2012	2013
Mixed water	1^{st} orchard	12.2	11.2	1.31	1.26	9.31	8.67	3.40	3.15	91	111
	2^{nd} orchard	12.6	11.9	1.09	1.38	11.6	8.62	3.35	2.91	123	112
	3^{rd} orchard	12.7	11.9	1.18	1.29	10.8	9.29	3.54	3.22	114	135
	Mean	**12.5**	**11.7**	**1.19**	**1.31**	**10.5**	**8.86**	**3.43**	**3.09**	**109**	**119**
Fresh water	4^{th} orchard	9.12	9.71	1.65	2.16	5.53	4.50	3.25	3.22	115	134
	5^{th} orchard	10.4	9.50	1.42	1.09	7.32	8.72	3.50	3.65	86	103
	6^{th} orchard	12.4	12.1	1.88	1.45	6.58	8.34	3.10	3.01	110	105
Mean		**10.6**	**10.4**	**1.65**	**1.57**	**6.48**	**7.19**	**3.28**	**3.29**	**104**	**114**
Significance^{z}											
Water source		*	*	*	*	*	*	NS	NS	NS	NS
Orchard		*	*	NS	NS	NS	NS	NS	NS	NS	NS
Water source* orchard		NS	NS	NS	NS	NS	NS	NS	NS	NS	NS

NS: not significant; * significant at 0.05 level.

The results of the fruit quality in first and second seasons presented in Table 4 revealed that using mixed water resulted in significant changes in fruit chemical compositions. Soluble solid contents increased and titratable acidity % decreased, that lead to a significant increase in the Brix/acid ratio. Earlier reports revealed that

SSC content increased in citrus fruit irrigated with wastewater [7] and also in tomato irrigated with high saline water [34]. On the other hand, significant differences in SSC among orchards in both seasons indicated that there are other factors affecting fruit SSC in addition to the water source. Using mixed water in irrigation did not produce significant effects on Juice pH and total ascorbic acid content.

3.3.2. Elements Concentration in Fruit Peel and Pulp

Table 5. showed the mean value of elements and heavy metals concentration (mg.100gm^{-1} fresh weight) in the peel and pulp of Navel orange fruit affected by MW and FW irrigation. All metal concentrations were several fold higher in fruits collected from MW irrigated orchards compared to FW irrigated ones. The mean values of K, Fe, Zn and Na in fruit irrigated with MW were two or more times higher, especially for iron which was 7 times of its concentration in FW irrigated fruits. In addition, Mn and Ni had been detected only in plants irrigated with mixed water, while Cu and Cd are not detected with both sources of water either in fruit peel or pulp. Data presented declared that the mixed water irrigation had a significant effect on the elements concentration in Navel orange fruit. In this respect, [33,35] found significant increase in the concentrations of Fe, Mn, Zn, Cu, Ni, and Cd in wastewater irrigated plants as compared with FW irrigated plants. Also [36,37,38]; proved higher absorption of some risk elements by various crops irrigated with municipal wastewater.

Table 5. Mean values of element concentrations in Navel orange fruit affected by two irrigation sources

Water source	Part of fruit	K	Fe	Mn	Cu	Zn	Ni	Cd	Na
					(Mg.100gm^{-1} fresh weight)				
Mixed water	Peel	761	11.21	0.29	nd	4.98	nd	nd	58.1
	Pulp	1364	10.24	1.85	nd	6.80	0.275	nd	158
	Mean	**1063**	**10.73**	**1.07**	**nd**	**5.89**	**0.138**	**nd**	**108**
Fresh water	Peel	569	2.88	nd	nd	1.52	nd	nd	17.4
	Pulp	835	nd	nd	nd	1.52	nd	nd	46.4
	Mean	**702**	**1.44**	**nd**	**nd**	**1.52**	**nd**	**nd**	**31.9**
Significance									
Water source		*	*	*	-	*	*	-	*
Part of fruit		*	NS	*	-	NS	*	-	*
Water source* part of fruit		NS	NS	*	-	NS	*	-	*

nd: not detected; NS: not significant; * Significant at 0.05 level.

The concentrations of K, Mn, Ni and Na in fruit pulp were significantly higher than in the peel. On the other hand, Fe and Zn had no significant differences within fruit peel and pulp. These results indicated that heavy metal accumulation differed according to the part of the fruit. These findings are in harmony with [6] who recorded different level of heavy metals accumulation in the edible parts of different kinds of plants, irrigated with the same wastewater.

The concentrations of all elements in fruit peel and pulp were not in the same level in the irrigation water or in the soil, this could be explained by the fact that heavy metals are taken up by the roots and only smaller amounts were translocated to the other parts of the plant [39].

4. Conclusion

Mixed water had a high nutritive value that may be useful in the reduction of fertilizers applied to orchards. Using mixed water in irrigation for a long time affected the physical and chemical properties of the soil. Soils irrigated with mixed water revealed a significant increase in soil pH, Ec, and elements concentration. On the other hand, using MW increased yield parameters of Navel orange and improved some fruit quality parameters. Fruit still looked apparently healthy despite increasing of K, Fe, Mn, Zn, Ni and Na in both of peel and pulp. Thus, using waste water irrigation may cause a potential risk to soil quality and productivity. In addition, there is a risk associated with consumption crops irrigated with mixed water for a long-term.

We must be careful about using mixed water in crops irrigation. Therefore, regular monitoring of metals level, in soil and agricultural products is necessary to protect soil properties and minimize hazards from toxic contaminants in agricultural products and the food chain. Furthermore, reduction of fertilizer applications is essential to protect soil and crops from trace elements accumulation.

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